QUALITY ASSURANCE PROJECT PLAN

OPERABLE UNIT 2
McINTOSH, ALABAMA

Prepared for:

OLIN CORPORATION

Prepared by:

MACTEC ENGINEERING AND CONSULTING, INC.
KENNESAW, GEORGIA

June 30, 2008
Revised October 9, 2008

Project No. 6100-08-0035

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OLIN CORPORATION

Charleston, Tennessee

MACTEC Engineering and Consulting, Inc.

Kennesaw, Georgia

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Project Number 6100-08-0035

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TABLE OF CONTENTS

LIST OF TABLES

Table

LIST OF FIGURES

Figure

LIST OF APPENDICES

Appendix

ABBREVIATIONS AND ACRONYMS

AA	Atomic Absorption
ADEM	Alabama Department of Environmental Management
AES	Analytical Environmental Services, Inc.
AOC	Administrative Order of Consent
ASTM	American Society for Testing and Materials
AVS/SEM	Acid Volatile Sulfide/Simultaneously Extracted Metals
AWQC	Ambient Water Quality Criteria
Basin	Olin Basin
Battelle	Battelle Marine Sciences Laboratory
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CHMM	Certified Hazardous Materials Manager
COC	Constituent of Concern
CPR	Cardiopulmonary Resuscitation
DDD	Dichlorodiphenyldichloroethane
DDE	Dichlorodiphenyldichloroethylene
DDT	Dichlorodiphenyltrichloroethane
DDTR	2,4'- and 4,4'- DDD, DDE, and DDT
DOC	Dissolved Organic Carbon
DQI	Data Quality Indicator
DQO	Data Quality Objective
DSA	Data Quality Assessment
ECD	Electron Capture Detector
EDD	Electronic Data Deliverable
ESPP	Enhanced Sedimentation Pilot Project
ERA	Ecological Risk Assessment
GC	Gas Chromatograph
GPC	Gel Permeation Chromatography
HASP	Health and Safety Plan
ICP-AES	Inductively Coupled Plasma-Atomic Emission Spectroscopy
LCS	Laboratory Control Sample

ABBREVIATIONS AND ACRONYMS (Continued)

MACTEC	MACTEC Engineering and Consulting, Inc.
MDL	Method Detection Limit
MDR	Mixed Diamine Reagent
mm	millimeter
MS	Mass Spectroscopy
MS/MSD	Matrix Spike/Matrix Spike Duplicates
NCP	National Contingency Plan
ng/L	nanograms per liter
NIST	National Institute of Standard Testing
NPDES	National Pollutant Discharge Elimination System
NPL	National Priority List
Olin	Olin Corporation
ORP	Oxidation Reduction Potential
OSHA	Occupational Safety and Health Administration
OU-1	Operable Unit 1
OU-2	Operable Unit 2
PACE	Pace Analytical Laboratories
PARCC	Precision, Accuracy, Representativeness, Completeness, Comparability
PE	Professional Engineer
PM	Project Manager
POC	Point of Contact
QA	Quality Assurance
QAM	Quality Assurance Manual
QAPP	Quality Assurance Project Plan
QC	Quality Control
QL	Quantitation Limit
RCRA	Resource Conservation and Recovery Act
RI/FS	Remedial Investigation/Feasibility Study
RL	Reporting Limit

ABBREVIATIONS AND ACRONYMS (Continued)

RPD	Relative Percent Difference
SHSO	Site Health and Safety Officer
SOP	Standard Operating Procedure
TOC	Total Organic Carbon
TSS	total suspended solids
USEPA	United States Environmental Protection Agency
WCC	Woodward-Clyde Consultants
WP	Work Plan
WTP	Work and Test Procedure

QUALITY CONTROL SIGNATURE PAGE

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Tables 2-2, 2-3, 2-4, 2-5	JAH	WPB
Figure 1-1	JAH	WPB
Figures 1-2, 1-3	APS	FKM

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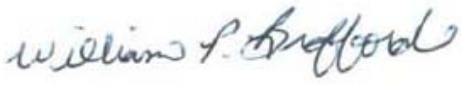

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1.0 PROJECT – TASK ORGANIZATION

Olin Corporation (Olin) has contracted with MACTEC Engineering and Consulting, Inc. (MACTEC) to prepare this Quality Assurance Project Plan (QAPP) to perform treatability studies and investigative activities as part of a Remedial Investigation/Feasibility Study (RI/FS) at the Olin McIntosh Operable Unit 2 (OU-2) located in Washington County, Alabama. This QAPP has been developed to outline the procedures and methodologies that will be used to document the quality of the sampling and analytical data collected at OU-2 and may be used for other environmental activities at McIntosh Plant. This QAPP addresses the quality assurance/quality control procedures for the RI/FS studies currently being performed and activities anticipated in the future. The QAPP is written in the present tense.

Sampling and analysis activities at OU-2 include but are not limited to:

- Groundwater samples collected from monitoring wells;
- Soil samples;
- Surface water and/or sediment samples collected from the Olin Basin (Basin), Round Pond, Tombigbee River and adjacent areas; and
- Biota samples.

The RI/FS activities prior to 2006 were performed under the *Remedial Investigation (RI/Feasibility Study (FS) McIntosh Plant Site Work Plan*, dated December 1990, which incorporate quality assurance and quality control criteria.

This QAPP has been prepared in accordance with *EPA Requirements for Quality Assurance Project Plans for Environmental Data Operation EPA QA/R-5*, (U.S. Environmental Protection Agency [USEPA], 1994), *EPA Requirements for Quality Assurance Project Plans EPA/240/B-0/003 QA/R-5*, (USEPA, 2001), *Guidance for the Data Quality Objectives Process, EPA QA/G-4, EPA/600/R-96/055*, (USEPA, 2000), and *Guidance for Data Quality Objective Process for Hazardous Waste Sites, EPA QA/G-4HW, EPA/600/R-00/007*, (USEPA, 2000) per Section X.A of the Olin McIntosh Consent Order (USEPA, 1990). The organization of this QAPP follows the 2001 USEPA *Requirements for Quality Assurance Project Plans, EPA/240/B-0/003 QA/R-5*, and the USEPA *Region 4 Quality Assurance Section QAPP Pre-Screening Checklist 2005*.

Work plans (WPs) that have been submitted to EPA from 2006 to date include the following:

- Enhanced Sedimentation Pilot Project Baseline and Evaluation Sampling Plan, April 2006
- Storm Event Surface Water Sampling Plan, February 5, 2008
- Treatability Study Work Plan Cap Material Assessment, April 1, 2008
- Groundwater Investigation Work Plan, May 2, 2008

Specific Data Quality Objectives (DQOs) for specific work tasks are presented in Section 1.7 and provided in the associated sampling or test plan. If future sampling plans cover tasks outside those anticipated and provided in this QAPP, addenda will be provided as appropriate. EPA approval of this QAPP and associated WPs is required prior to implementation.

1.1 PROJECT ORGANIZATION

Olin is providing a team of professionals, including the assistance of subcontractors, to complete the work assignments in accordance with the procedures described in associated WPs and this QAPP. Olin has retained MACTEC Engineering and Consulting, Inc. (Kennesaw, Georgia) as a primary consultant for the OU-2 RI/FS. An organizational chart and flow of responsibilities is shown in Figure 1-1, and a brief description of Olin and MACTEC's responsibilities is listed below. Olin reserves the right to designate alternate personnel to fill project roles as required without officially modifying the QAPP. EPA will be notified of key project management personnel changes. The current team and their responsibilities are as follows:

1.1.1 Project Manager

The Olin Project Manager, Keith Roberts, is responsible for coordinating Olin's consultants, overall project management, supervising fieldwork, and evaluating data. Mr. Roberts is the primary point of contact for the USEPA and Olin's subcontractors. He also is responsible for projecting resource needs and facilitating the assignment of those resources to the project. The MACTEC Project Manager (PM), Cynthia Draper, is responsible for overall project scope, organization, schedule, budget, and quality for those activities assigned by Olin's Project Manager.

1.1.2 Project/Technical Principal

The Project/Technical Principal is responsible for the project's technical quality. Mr. Mike Bellotti is a Principal Geologist and is Olin's Technical Principal for the OU-2 groundwater investigation.

Dr. James R. Wallace is MACTEC's Project Principal, and Mr. Steve Youngs is MACTEC's Design Principal for this project. The Principals' responsibilities are to assure that each task meets the project's technical quality objectives.

1.1.3 Plant Principal Environmental Specialist

Ms. Toni Odom is Olin McIntosh Plant's Principal Environmental Specialist for the McIntosh Plant. Ms. Odom works with Olin's subcontractors to coordinate work performed within OU-2 and with resources at the McIntosh Plant.

1.1.4 Discipline Leaders

Discipline leaders are utilized to implement specific tasks. The discipline leaders work directly with the Project Manager and Project Principal to implement the assignment and prepare project deliverables.

- **Site Manager/Field Coordinator**

  The Site Manager/Field Coordinator manages and coordinates field activities. The Site Manager/Field Coordinator works directly with the PM to assist in the prioritization of scheduling tasks for investigation, interpretation of the data collected, and preparation of reports in addition to site management duties. The Site Manager/Field Coordinator is identified in the task-specific WPs.

- **Chief Environmental Engineer**

  Mr. Phil Pauquette is MACTEC's Chief Environmental Engineer. Mr. Pauquette is the overall technical and quality leader for environmental activities. His responsibilities include marketing, project execution, training and technical development, and quality assurance.

- **Quality Assurance**

  The Quality Assurance (QA) Officer, Mr. Paul Brafford, Certified Hazardous Materials Manager (CHMM), MACTEC, is responsible for the overall quality of the fieldwork associated with the work, and the quality of the chemical data generated. The QA Professional is independent from the PM and works directly with the Project Principal to monitor that the work being performed follows Olin's QA policies and the QA

requirements of the project. The QA Officer may issue a Stop Work Order if appropriate corrective action has not been implemented and the non-conformance is considered quality affecting. Specific responsibilities include:

- Review of analytical protocols for measuring and monitoring;
- Review of the laboratory personnel qualifications, equipment, facilities, and analytical procedures prior to receiving samples;
- Review of the Quality Assurance/Quality Control (QA/QC) results with laboratory QA staff;
- Laboratory QC evaluations and review of corrective action recommendations (if required);
- Documents that appropriate QA/QC procedures have been established and are being implemented by the analytical laboratory and project personnel.
- Conducts surveillances of the field and/or laboratory activities, as appropriate.

- **Health and Safety Site Officer**

  A Site Heath and Safety Officer (SHSO), generally the Site Manager/Field Coordinator, is designated from the work crew assigned to each field task, and serves as the on-site resource for health and safety issues or concerns and administering the site specific Health and Safety Plan (HASP).

- **Team Engineers/Scientists**

  The Team Engineers for the Olin McIntosh OU-2 include a Design Engineer and a Treatability Engineer. The Design Engineer supervises activities related to the berm and gate design and the design of mechanical sampling systems. The Design Engineer is responsible for review and oversight of the contractor and procedures, assurance that goals are being met by the process, and review of plans and reports related to engineering design. The Treatability Engineer supervises activities related to the treatability study. Other Team Scientists may include a project biologist and ecological specialist, chemists, and risk assessors.

- **Project Chemist**

  The Project Chemist will supervise activities related to the chemical analysis, and chemical data quality and is responsible for reviewing and documenting the data validity. The Project Chemist will work with the PM and the QA Officer. Specific responsibilities include:

  - Production of the QAPP with assistance and input from other project personnel;
  - Tracking sample chain-of-custody through the laboratory;
  - Verifying that laboratory QC and analytical procedures are being followed as specified in the QAPP and reviewing sample and QC data. This review includes examination of raw data such as chromatograms and checking arithmetic calculations

for the samples analyzed, and inspection of reduced data, calibration curves and laboratory extraction logbooks;

- Producing and/or reviewing a detailed validation report of the data collected.

- **Database Manager**

  The Database Manager is responsible for the management, specification, and importing of electronic data deliverables (EDDs) into an Olin master database. The Database Manager produces task-specific queries and tables of validated data for use in project assessments and deliverables. The Database Manager works with the PMs to provide consistent and correct data to support Olin projects.

1.1.5 Laboratory

Three laboratories currently receive and analyze samples collected at OU-2. The Olin PM determines which laboratory is used on a case-by-case basis. Additional laboratories may be added to meet project requirements without official modification of the QAPP. EPA will be notified of laboratory changes in the Monthly Progress Report. The current laboratories and their point of contact (POC) are presented below.

- Analytical Environmental Services, Inc. (AES) (Atlanta, GA) – responsible for chemical analytical testing of ISCO samples from the Basin.

  Justin Sasser, PM (TSS analysis)
  Analytical Environmental Services, Inc.
  3785 Presidential Parkway
  Atlanta, GA 30340-3704
  770.457.8177
  770.457.8188 (fax)

- Pace Analytical Services (PACE) (St. Rose, LA and other locations) – responsible for chemical analytical testing of samples collected from groundwater, surface water, soil, sediment, and biota sampling programs.

  Tod Noltemeyer, PM (biota analysis)
  PACE Analytical
  25 Kessel Court, Suite 105
  Madison, WI 53711
  608.232.3300 ext. 302
  Tod.Noltemeyer@pacelabs.com

  Cindy Olavesen, PM (lab analysis)
  PACE Analytical
  1000 Riverbend Blvd. Suite F
  St. Rose, LA 70087
  504.469.0333
  504.469.0555 (fax)
  Cindy.Olavesen@pacelabs.com

  Biota samples will be shipped to:
  PACE Analytical
  1241 Bellevue Street, Suite 9

Green Bay, Wisconsin 54302

- Battelle Marine Sciences Laboratory (Battelle) (Sequim, WA) – responsible for chemical and physical analytical testing of samples collected from OU-2. Battelle analyses include low-level mercury, methylmercury, and acid volatile sulfide/simultaneously extracted metals (AVS/SEM) analyses.

Brenda Lasorsa, PM
Battelle Marine Sciences Lab
1529 West Sequim Bay Road
Sequim, WA 98382
360.683.4151 x13650 (main)
360.681.3650 (office)
360.681.3640 (lab)
360.681.3699 (fax)
brenda.lasorsa@pnl.gov

The laboratories are responsible for:

- Receiving samples from the field and verifying that incoming samples correspond to the completed chain-of-custody form;
- Maintaining records of incoming samples. Tracking samples through processing, analysis, and appropriate disposal at the conclusion of the program;
- Informing the Field Sampling Coordinator and Analytical Laboratory POC of discrepancies between chain-of-custody forms and sample package contents;
- Reviewing raw data with laboratory analysts by comparison to calibration and quality control (QC) records; and
- Preparing analytical data including QC data for validation by the Quality Assurance Officer.

1.1.6 Subcontractors

Subcontractors may be used by Olin to perform specific project elements. Subcontractors to be used are screened by Olin's PM or Olin's designee and selected based on their qualifications.

1.1.7 Project Schedule

Specific assignment work schedules are developed for each task on the basis of the DQOs and are described in the task-specific WPs, which begin following approval of the WP by the, USEPA.

1.2 PROJECT BACKGROUND

Olin is conducting a Remedial Investigation/Feasibility Study (RI/FS) at its McIntosh, Washington County, Alabama Plant Site (site) under the oversight of the United States Environmental Protection Agency (USEPA). Olin signed an Administrative Order of Consent (AOC), effective May 9, 1990, to satisfy the National Contingency Plan (NCP; 40 Code of Federal Regulations 300). The site is an active chemical production facility, located approximately 1 mile east-southeast of the town of McIntosh, Washington County, Alabama. The site is listed on the National Priority List (NPL) of Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) and is composed of two operable units (OUs): Operable Unit 1 (OU-1), which consists of the manufacturing process area of the site, and OU-2, which consists of mostly wetlands adjacent to the Tombigbee River.

Numerous studies have been conducted at the site. Reports on these studies include an RI report (Woodward-Clyde Consultants [WCC], 1993), FS report (WCC, 1993), additional ecological studies to supplement the RI (WCC, 1994a), an Ecological Risk Assessment report (WCC, 1995), a second FS report (WCC, 1996), OU-2 Remedial Goal Option Support Sampling Report (URS, 2002), and the Enhanced Sedimentation Pilot Project Baseline Sampling Report ([MACTEC], 2007a). In 1994 and 1995, Olin collected additional data to better assess ecological risks and more adequately evaluate remedial alternatives for OU-2 (WCC, 1994a). These data were reported in a study report (WCC, 1994b) and incorporated into an Ecological Risk Assessment (WCC, 1995) and OU-2 Feasibility Study (WCC, 1996).

The FS and implementation of the remedial action have been completed for OU-1 and is being monitored under the Resource Conservation and Recovery Act (RCRA) program. Work at OU-2 is ongoing.

1.3 OU-2 DESCRIPTION

OU-2 comprises the Basin, Round Pond, surrounding wetlands on the Olin property, and the former wastewater ditch that discharged to the Basin from 1952 to 1974 (Figures 1-2 and 1-3). The Basin and Round Pond cover approximately 74 and 4 acres, respectively. The Basin is located between the bluff to the west and the Tombigbee River to the east. The bluff is approximately 20 to 30 feet higher in elevation than the floodplain area near the Basin. The Basin and Round Pond are thought to be part of a natural oxbow lake lying within the floodplain of the Tombigbee River.

1.4 HISTORY

Mercury has been detected in sediment samples collected at OU-2 but not in filtered surface water samples (MACTEC, 2007a). The primary release mechanism for mercury to OU-2 was the discharge through the former wastewater ditch from 1952 to 1974 (WCC, 1993). Surface runoff and treated wastewater from the plant have not been discharged to the Basin since 1974. The plant effluent and stormwater discharge are permitted and monitored under the National Pollutant Discharge Elimination System (NPDES). The current discharge is acceptable within the NPDES limits.

With the conditional approval of the USEPA (USEPA, 2005), a berm and gate system for an ESPP was constructed by Olin between June 2006 and March 2007 (Figure 1-3). The ESPP includes construction of a berm and gate system to capture floodwaters and the associated sediment so that sedimentation in the Basin is enhanced. This ESPP is a treatability study being performed under the FS. The effectiveness of the ESPP is currently being monitored during a three-year demonstration period.

1.5 REGULATORY STATUS

Previous ecological studies in the OU-2 Basin (WCC, 1994b; 1995) have demonstrated potential ecological risk associated with mercury concentrations in sediments. The primary constituent of concern (COC) in sediments and biota is mercury. Inorganic mercury could undergo methylation in sediments to form the more biologically active methylmercury. Other COCs include hexachlorobenzene and the isomers of dichlorodiphenyl-trichloroethane [DDT], dichlorodiphenyldichloroethylene [DDE], and dichlorodiphenyl-dichloroethane [DDD]..

As part of the proposed remedial action to reduce potential ecological risk, Olin implemented an ESPP, which consisted of constructing a berm with a gate around OU-2 to trap floodwaters with suspended solids from the Tombigbee River during flood events, thereby increasing sedimentation and enhancing natural capping of the sediments. The berm and gate system became operational in March 2007; physical features and components of OU-2 and the berm/gate system are depicted in Figure 1-3. Baseline physical and chemical data were collected to document conditions prior to implementation of this remediation strategy. In addition, annual samples will be collected over the three-year ESPP evaluation period to assess the effectiveness of the enhanced sedimentation as a remediation alternative.

In addition to the ESPP, additional WPs have been submitted to USEPA. These include treatability studies for alternative in situ caps (MACTEC, 2008a), storm event sampling (MACTEC, 2008b), mercury methylation research (MACTEC, 2007b), and a groundwater investigation WP (MACTEC, 2008c). An expansion of the annual ESPP sampling is also planned. These WPs and sampling plans are collectively referred to as "work plans" in this QAPP.

1.6 PROJECT OBJECTIVE

The QAPP provides the necessary QA/QC procedures that site studies are performed in accordance with acceptable protocols, and that the data generated meet the overall project objectives for precision and accuracy. This QAPP provides traceable sampling and analysis procedures, personnel requirements, chain-of-custody and documentation requirements, and specific criteria for determining data acceptability. The QAPP also establishes the procedures to address data deficiencies, data reduction and evaluation, and preparation of field study reports, which will be produced so that outputs are accurate and technically reasoned. Separate Sampling and Analysis Plans and/or WPs are prepared on a case-by-case basis. Precision, accuracy, representativeness, comparability, completeness (PARCC), and sensitivity are assessed before data are used for characterization or risk assessment purposes.

The objectives of the data collection include the evaluation of risk, the update of the ecological risk assessment (ERA), defining the remediation goals protective of human health and the environment, support for the evaluation of alternatives for remedial decision making, collection of data to confirm the delineation of the constituents of concern and demonstrate the effectiveness of the ESPP and other environmental activities.

1.7 QUALITY OBJECTIVES AND CRITERIA FOR MEASUREMENT DATA

1.7.1 Data Quality Objectives

Data Quality Objectives (DQOs) are used to determine the type, quantity, and quality of data needed to reach defensible decisions. DQOs define the performance criteria and are part of a systematic planning process. This framework is used so that the level of detail is commensurate with the intended data use and available resources. Specific DQOs for specific work tasks are provided in the associated sample or test plan. A seven-step process has been developed by USEPA (as outlined in the *Guidance for the Data Quality Objectives Process, EPA QA/G-4, EPA/600/R-96/055* (August, 2000a) and the *Guidance for Data*

Quality Objective Process for Hazardous Waste Sites, EPA QA/G-4HW, EPA/600/R-00/007 (January, 2000b). Each step is summarized below.

1.7.1.1 Step 1 – State the Problem

In Step 1, an effective planning team is assembled, the problem is described, a conceptual model is developed, and budget and schedule constraints are outlined.

Project/Planning Team – A multidisciplinary team includes technical staff and decision makers from Olin and the USEPA. The project organization for decision making is provided in Section 1.1 and presented in Figure 1-1.

Problem - The ecological risk assessment for OU-2 (WCC, 1995) identified potential ecological risk associated with OU-2 sediments. The COCs in sediments and biota were identified as mercury, hexachlorobenzene, and 2,4'- and 4,4'- DDD, DDE, and DDT (DDTR). In addition, the USEPA is concerned that mercury in sediments within the Basin and Round Pond and in wetland soils may be transported to groundwater in and around OU-2. Olin has submitted WPs to evaluate the ESPP as means to cap sediment, evaluate alternative cap materials, and investigate groundwater. The activities to delineate the environmental conditions are presented in the WPs previously submitted.

Conceptual Site Model –A conceptual site model for groundwater is presented in Section 3.0 of the Groundwater Investigation WP (MACTEC, 2008c). This model is further refined in the updated ERA. Other CSMs will be developed to address pathways other than groundwater, as appropriate.

Resources/Schedule – Resources and relevant deadlines are committed as appropriate to meet commitments to the USEPA.

1.7.1.2 Step 2 – Identify the Goal of the Study

In Step 2, the principal study question or decision is identified and options for addressing the study (i.e. alternative actions or outcomes) are defined. These two elements are then combined to develop a decision statement that must be resolved using data for defensible environmental decision making. For complex investigations, multiple decisions are possible.

Decision Statement - The primary goals of the RI/FS for OU-2 is to characterize OU-2 in terms of ecological risk, select appropriate clean up goals, and collect data to support a FS. This is currently being addressed through the implementation of the ESPP (MACTEC, 2006), the Groundwater Investigation WP (MACTEC, 2008c), the methylation of mercury research plan (MACTEC, 2007b), Storm Event Sampling Plan Addendum (MACTEC, 2008b), and the Treatability Study (MACTEC, 2008a). Additional studies needed to support the primary goal of the RI/FS at OU-2 will be subject to this QAPP and EPA approval.

Principal Study Questions – Each WP presents specific study questions pertinent to the task being performed.

Alternative Actions – The alternative actions may include recommending no further action, re-evaluation of remedial alternatives, or collecting additional data to support the selection and implementation of a final remedy.

1.7.1.3 Step 3 – Identify Information Inputs

In Step 3, data requirements and sources are identified along with sampling and analysis methods and other informational inputs that will be required to resolve the decision and to determine which inputs require environmental measurements. Action levels for constituents are also determined based upon the informational inputs.

Information Required to Resolve the Decision Statement – Data includes the analyses of the identified COCs in the impacted media or media that may become impacted. The main COC in sediments and biota is mercury. Inorganic mercury could undergo methylation in sediments to form the more biologically active methylmercury. Other COCs include DDTR and hexachlorobenzene. Data is required to support the on-going WP tasks which include the ESPP, treatability studies for alternative caps (MACTEC, 2008a), storm event sampling (MACTEC, 2008b), mercury methylation research (MACTEC, 2007b) and a groundwater investigation WP (MACTEC, 2008c). Additional WPs beyond those listed in the QAPP, may be prepared to support the primary goals of the RI/FS. Data inputs will be provided in these future WPs for review and approval by EPA.

Probable parameters to be analyzed by media are summarized in Table 1-1 with the laboratory analytical methods as presented in Table 1-2. The rationale for the parameters to be collected and method selection is also presented in Table 1-2. Both screening and definitive data are collected and used.

- ESPP: The plan to evaluate the effectiveness of the ESPP in limiting exposure to sediments and reducing risk to the environment uses a three-pronged approach. Specifically, this approach includes: 1) chemical analysis of the surface water and sediment to document changes in chemical characteristics; 2) methods to document sediment accumulation (total suspended solids [TSS] analysis, sediment pins, sediment traps, and bathymetric surveys); and 3) bioaccumulation cage studies which document the effect of ESPP on biota representative of OU-2. In addition, annual samples are collected over the three-year ESPP evaluation period to assess the effectiveness of the enhanced sedimentation as a remediation alternative.

- Storm Event Surface Water Sampling: The purpose of the storm event sampling activities is to collect surface water samples that aid in evaluating the solids load into and within the Basin during various storm events. The solid load data from the floodwaters are used to assess the amount of sediment available for accumulation in the Basin.

- Treatability Study WP Cap Assessment: The purpose of this WP is to present the technical approach for the evaluation of potential cap materials suitable for covering OU-2 Basin sediments should the ESPP results be unfavorable. The objective of the treatability study is to observe and document the degree of re-suspension, the potential for intermixing or entrainment of sediment in cap materials, and the potential to increase available mercury for biotic uptake during placement of cap materials.

- ISCO Sampling: ISCO samples have been collected from the Olin dock and the Ciba dock on the Tombigbee River since June 2004. The TSS data collected from samples give an understanding of potential sediment load to OU-2 from the Tombigbee River throughout flood events.

- Groundwater Sampling: The purpose of this WP is to evaluate the potential for mercury to migrate to and/or from groundwater at OU-2.

- Mercury Methylation Research (2008c): This report presents the results of the mercury methylation research and along with a description of both historical and baseline indicator parameters applicable to OU-2.

Source of Required Information – In addition to the new physical and chemical data acquired from each of the samples collected and analyzed, additional sources of information include data collected prior to and during the RI (WCC, 1993), FS (WCC, 1995), OU-2 Remedial Goal Option Support Sampling Report (URS, 2002), and ESPP Baseline Study (MACTEC, 2007a).

Establish Action Levels – Ambient Water Quality Criteria (AWQC) are the action levels utilized for decision making when reviewing analytical results for surface water. An effort has been made to select the appropriate method to obtain data at or below the action levels. AWQC are presented in Table 1-3. No action levels have been established for sediment or biota. Action levels for sediment or biota will be based upon the results of the updated ERA. Therefore, in the absence of site specific action levels, analytical methods with the lowest reasonably achievable MDLs were selected for analysis of COCs in sediment and biota.

Analytical Methods to Provide the Necessary Data – The analytical and physical testing methods to obtain the information required are described in Section 2.4 of this QAPP and in Table 1-3.

1.7.1.4 Step 4 – Define the Boundaries of the Study

In Step 4, the target population, geographic boundaries, time frame for data collection, and scale of the decision making process are defined. Practical constraints to data collection are also addressed at this stage.

The target population involves both continuous media (surface water, groundwater, sediment, and soil) as well as biota (fish and clam samples). The sample volumes to be collected are defined based on the analytical method requirements (Table 1-4). The geographic boundaries include OU-2. The temporal boundaries or time frame for sample collection is presented in each of the task specific WPs. The rationale of when to collect samples is discussed relative to representativeness and may include collection during storm/flood events or during non-fluctuating water levels. Individual WPs include sample design specifics that present the sample volumes and dimensions that are appropriate to observe the patterns on the scale of interests. Samples are collected and shipped to the laboratory within recommended holding times. Each study considers practical constraints regarding safety, ecological activity, flood events, analytical holding times, sample volumes, and the potential need to sample for low-level mercury.

1.7.1.5 Step 5 – Develop the Decision Rule

In Step 5, parameters are compared to corresponding criteria or action levels assuming perfect information. A decision rule in the form of an "If…then…" statement is developed.

The Statistical Parameter that Characterizes the Population of Interest – Depending on the work task element, a statistical evaluation may be performed for the COCs. Statistical evaluations may include determination of the data distribution, upper 95 percent confidence limits about the mean, and maximum values.

The Action Level for the Decision – Depending on the work task element, the action level may include comparison to a screening value, a physical characteristic, a timed event, or a volume requirement. As previously mentioned, the action level for surface water is the Alabama Department of Environmental Management AWQC.

The Action Levels Exceed Measurement Detection Limits – The analytical methods selected obtain data at or below the action levels for water samples. Mercury is analyzed by EPA Method 1631E to achieve a reporting limit below the AWQC of 12 nanograms per liter (ng/L) and EPA Method 8081A was selected to report hexachlorobenzene and DDTR near their AWQC of 0.0003 micrograms per liter (µg/L) and 0.001µg/L, respectively. In addition, low-level sampling for mercury via Method 1669 is utilized to collect surface water, and groundwater samples. The environmental sampling is designed with sufficient replicates and quality assurance/quality controls (QA/QC) so that environmentally significant effects can be qualified. Analytical methods to meet this limit are listed in Table 1.3.

Decision Rule – *A decision rule in the form of an "If…then…" statement is developed*. For example, mercury concentrations in groundwater are screened based on the ambient water quality criterion. If this screening level is exceeded, then an appropriate model may be prepared to determine a concentration of concern. If groundwater concentrations exceed this concentration of concern, then additional assessment may be performed to determine the nature and extent of mercury and other COCs. Each WP will describe the decision rules as appropriate.

1.7.1.6 Step 6 – Specify Performance of Acceptance Criteria

In Step 6, perfect information is no longer assumed. Recognizing that decisions are made on a sample data set which represents a small part of the larger population and is subject to errors, numerical values are considered in attempt to minimize decision errors. The purpose of Step 6 is to specify quantitative performance goals (probabilities) for limiting uncertainty in the data. These probabilities represent the amount of uncertainty considered tolerable.

Each WP will describe the performance acceptance criteria.

1.7.1.7 Step 7 – Develop the Plan for Obtaining the Data

The purpose of Step 7 is to develop a cost-effective sampling and analysis program. Historical data were and continue to be reviewed. Parameters were selected based on the results of previous studies and the large quantity of existing data. In addition, a reconnaissance was undertaken to select specific sample locations with suitable media for sampling. The overall elements of the sampling program are documented in the WPs. Assumptions supporting the sampling program are provided in the WPs and supporting documents.

The number of samples and locations were selected after reviewing previous studies and the area to be studied. The analytical methods were selected so that the detection limits are low enough to compare data results to screening criteria where applicable. In this way, the sampling design attempts to minimize and manage these errors. Each WP presents the rationale for sampling location, the number of samples to be collected, and the media to be analyzed. Sample locations are based on the bathymetric studies, surface water depth, and previous data.

The overall data quality objective is to produce data of sufficient quality for use in risk assessment, to support remedial alternative selection, and to monitor the effectiveness of potential remedial alternatives. These policies are intended to provide analytical data that will yield comprehensive and valid results and comply with applicable federal and State regulations.

1.7.2 Data Quality Indicators and Criteria

The objective of this QAPP is to develop and implement procedures for field sampling, chain-of-custody, laboratory analysis, and reporting that provide results that are legally defensible and of sufficient quality to meet the DQOs. Specific procedures for sampling, chain-of-custody, laboratory instrument calibration, laboratory analysis, reporting of data, internal quality control, audits, preventive maintenance of field equipment, and corrective action are described in subsequent sections. Data usability can be determined by review of field and laboratory data quality indicators (DQIs). The DQIs are the QA elements necessary to document the quality of the chemical data. DQIs for chemical data are expressed in terms of precision, accuracy/bias, representativeness, completeness, comparability (PARCC), and sensitivity. QA objectives provide the mechanism for ongoing control and evaluation of data quality throughout the

project and ultimately are used to define the data quality achieved for the various measurement parameters. Duplicate, matrix spike/matrix spike duplicate, field blank, and equipment rinsate samples are used to evaluate the effectiveness of laboratory methods and the sample collection methods used by the field crew. The goal is to obtain 90 percent completeness of the data.

The laboratory DQI program is assessed through internal laboratory quality control (QC), including method blanks; laboratory control samples (LCSs), surrogate standards, internal standards, and calibration standards. .

The field DQI program is performed so that the samples being collected are representative of the media being sampled, and that the data generated are valid. This is accomplished through:

- Collection of adequate number and type of sample from representative locations during the appropriate time frames.
- Use of the standard field procedures, also known as Work and Test Procedures (WTP);
- Accurate and detailed record keeping in the field notebooks and field logs;
- Proper calibration of field equipment according to manufacture's instructions; and
- Collection and analysis of QA samples potentially including field duplicates, rinsate blanks, trip blanks, and matrix spike/matrix spike duplicate (MS/MSD) samples.

The purpose of this section is to address the specific objectives for PARCC and sensitivity. These data quality criteria are discussed below.

1.7.2.1 Precision

Precision is a measure of the degree to which two or more measurements agree. One way to estimate precision is by calculation of relative percent difference (RPD) of duplicate analyses or duplicate spike analyses.

Field precision is assessed through the collection and measurement of field duplicates. A rate of 1 duplicate per 10 analytical samples was selected for the OU-2 projects. . Field precision goals for this project are 35 percent for water duplicates and 50 percent for soil/sediment/tissue duplicates. The field precision goals are based on laboratory estimates of precision derived from the laboratory's internal

QA/QC and an estimate of field precision. An added measure of precision is obtained by collecting QA split samples, which are field duplicate samples sent to a USEPA-designated laboratory for analysis. A split sample duplicate compares results from two different laboratories, ultimately deriving a determination of RPD for each constituent present. RPDs are calculated as shown below.

Precision in the laboratory is assessed through the calculation of RPDs for two or more replicate samples. The RPD equation is given by:

RPD = A - B (100 percent)/[(A + B)/2]
Where: RPD = Relative Percent Difference
A = First sample value
B = Second sample value

Laboratory precision is assessed at a rate of 1 per 20 analytical samples. Laboratory precision criteria are presented in Table 1.3.

1.7.2.2 Accuracy

Accuracy is the degree of agreement between an observed value and an accepted reference value. Accuracy in the field is assessed through the use of field and trip blanks and through the adherence to sample handling procedures, applicable preservation techniques, and holding times.

Laboratory accuracy is assessed through the analysis of matrix spikes or reference materials and the determination of percent recoveries. The equation to be used for accuracy is listed below.

$$R = [(A - B)/S](100 \text{ percent})$$

Where:

R = Percent Recovery
A = Value obtained by analyzing the sample with the spike added
B = Background value, *i.e.* the value obtained by analyzing the sample alone
S = Final concentration of the spike added to the sample

Accuracy control limits are presented in Table 1-3. Laboratory accuracy is assessed at a rate of 1 per 20 analytical samples.

1.7.2.3 Representativeness

Representativeness is the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition.

Representativeness of field data is dependent upon an adequate sampling program. Each WP presents the basis for the sampling design and how the design was developed so that the resulting data are adequately representative.

Representativeness in the laboratory is achieved using proper analytical procedures, meeting sample-holding times, and analyzing and assessing field-duplicated samples. The sampling program in each WP is designed to provide data representative of conditions at OU-2. During development of the sampling programs, existing analytical data, physical setting and processes, and constraints inherent to the sampling of the media of interest were considered.

1.7.2.4 Completeness

Completeness is a measure of the amount of valid data obtained compared to the amount that was collected. The equation for completeness is presented in below.

$$\text{Percent Completeness (\%)} = \frac{\text{(Number of accepted data points)} \times 100}{\text{Total number of samples collected}}$$

The completeness objective for field samples will be 85 percent or greater. A minimum completeness objective of 90 percent has been set for analytical samples.

1.7.2.5 Comparability

Comparability is the confidence with which one data set can be compared with another. Comparability of field data is dependent upon an adequate sampling program design and using proper sampling techniques and is satisfied by following the WP.

Analytical data are comparable when similar sampling and analytical methods are used and documented in accordance with the QAPP. Consideration is given to seasonal conditions, river flow, or other environmental factors that could influence analytical results.

1.7.3 Sensitivity

Sensitivity is the measure of the ability of the analytical methods or instruments to distinguish a low value from zero. Sensitivity can be measured by the attainment of method detection limits (MDLs), reporting limits (RLs), and/or PQLs. Method MDLs, RLs, and PQLs are reviewed and selected to meet the project DQOs. The MDL is the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the value is above zero. It can be established and maintained as described in 40 CFR Part 136 Appendix B (Federal Register, 1992). The RL is sometimes called the quantitation limit (QL) and is the lowest level that can be achieved within specified limits of precision and accuracy during routine laboratory operating conditions as defined by SW 846.

The proposed RLs meet the AWQC for mercury and methylmercury. The most current USEPA methods were selected for the analysis of hexachlorobenzene and DDTR.

Rinsate blanks, method blanks, field blanks, duplicates, MS/MSD and QC split samples can be used to measure affects on or to verify sensitivity. To the extent practical, the sampling and analytical systems must be free of contamination or interferences that impact detection limits and lower sensitivity. To measure and understand this effect, rinsate blanks, method blanks, field blanks, duplicates, and MS/MSDs are collected and analyzed with project samples. The analyses of QA split samples are also a tool to verify sensitivity.

Rinsate blanks consisting of distilled water poured over decontaminated sampling equipment are submitted to the analytical laboratory to assess the data quality from the field sampling program. Rinsate blanks are analyzed to check for procedural contamination during sampling. Rinsate blanks are only collected on non-dedicated sampling equipment. A rinsate blank is collected and analyzed per sampling device per media.

Method blank samples are generated within the laboratory and used to assess contamination resulting from laboratory procedures. Matrix spikes provide information about the effect of the sample matrix on the digestion and measurement methodology. Matrix spikes are performed in duplicate and are hereinafter referred to as MS/MSD samples. One MS/MSD will be analyzed for every 20 samples, or one per sample data group (SDG).

Field blanks pertain only to mercury and methylmercury surface water and groundwater analyses. Field blank samples are collected during surface water sampling for low-level (<5 parts per trillion) mercury (to detect atmospheric mercury that exists as ambient background). Two field blank samples are collected during surface water sampling; one in the morning and one in the afternoon. A jar containing organic-free water is used to fill a clean sample container in the field during surface water sample collection. For groundwater or water sampling for higher-level mercury (>0.20 parts per billion), one field blank is collected each day of sampling. Additional field blanks may be collected if weather or conditions change significantly.

Duplicate samples are analyzed to check for sampling and analytical reproducibility. One field duplicate is collected and analyzed for every 10 samples for surface water, groundwater, sediment, and soil. Field duplicate samples are not collected for tissue samples since several tissue samples are collected at each location. Instead, the laboratory prepares duplicate samples after sample homogenization but prior to analysis. Field duplicate precision goals are: 35 percent for water matrices and 50 percent for soil/sediment/tissue matrices.

1.8 TRAINING AND CERTIFICATIONS

Field personnel have appropriate Occupational Safety and Health Administration (OSHA) training for their job responsibilities in accordance with the Code of Federal Regulations (CFR), 29 CFR 1910.120. In addition, one individual per team has Cardiopulmonary Resuscitation and first aid training (as outlined in the HASP). Field personnel are also trained in low-level mercury sampling via EPA method 1669 and environmental sampling via the USEPA Region 4 Environmental Investigations Standard Operating Procedures and Quality Assurance Manual (EISOPQAM).

Training records and certifications are maintained by Olin and individual subcontractors. Personnel are required to review and update their training as needed. The Site Manager and SHSO review certifications of project personnel for compliance. If training is project-specific, the project PM notifies and arranges for personnel to acquire the necessary training to perform the work.

1.9 DOCUMENTATION AND RECORDS

The following information is included in each laboratory data report package.

1. Cover Letter with Laboratory Manager (or designee's) signature.
2. Data reports for each sample submitted which include at a minimum:
    - Results and reporting units for each parameter;
    - Project defined reporting limits;
    - Date of extraction(s) and analyses;
    - List of project specified methodologies for each parameter; and
    - Dates of sample collection and laboratory receipt.
3. Quality Control Summary Forms with method blank results, MS/MSD recoveries, and RPD calculations (where applicable).
4. Original Chain-of-Custody forms.
5. A Sample Receipt Record documenting the condition of the samples upon receipt by the laboratory.
6. A Case Narrative, as necessary, to discuss quality control limit exceedances, specific sample problems, and analytical methodology problems observed.

Field and laboratory records for this project are maintained for a minimum of 6 years after receiving the certification of completion by the USEPA per Section XIII of the Olin McIntosh Consent Order (USEPA, 1990).

1.10 DOCUMENT RETENTION

The following provides a general description of Olin's document retention policy:

1.10.1 Contractual Requirements

Files are maintained consistent with applicable contractual and/or legal requirements.

1.10.2 Retention of Files

Retention of files is in accordance with the requirements as specified in Section XIII of the Olin McIntosh Consent Order (USEPA, 1990) and includes:

- Records and documents which relate to the site will be preserved for a minimum of 6 years after the termination of the Consent Order.
- Olin will inform the USEPA within 90 calendar days prior to the destruction of any documents after the six-year period.

  This applies to all records and documents in Olin's possession, or the possession of their divisions, employees, agents, accountants, contractors, or attorneys. The Olin PM is responsible for overall implementation of this policy and for determining that disposal of individual files is appropriate. The Olin PM is also responsible for oversight of files and reports that are retained are properly documented and are stored in an organized and retrievable manner.

2.0 MEASUREMENT/DATA ACQUISITION

2.1 SAMPLING PROGRAM

The sampling programs include analyses for the parameters in Table 1-1. The sampling programs are described in the individual WPs. Mercury is the primary COC and is the focus of most studies. The remaining COCs, hexachlorobenzene, and DDTR are also included in this QAPP and are analyzed from select sample locations as identified in the WPs. Figures depicting the sample locations are presented in each task-specific WP.

The placement of sampling locations for investigations primarily uses the biased sampling approach. The project DQOs will aid in the determination of the number and media of samples to be collected for the appropriate data set. General criteria for the determination of sample location, number, size, and media are presented below.

- Sampling locations are selected based on historical data, access, reconnaissance, results from previous studies at OU-1 and OU-2, regional studies, bathymetric surveys, river characteristics, and project objectives.
- The number of samples and environmental media sampled are selected based on knowledge of OU-2, the area potentially affected, the conceptual site model, and/or will be statistically derived based on the overall decision rule.
- Biota samples are selected from indigenous species such as catfish, largemouth bass, and mosquito fish and/or native species such as freshwater mussels/clams. For example, as a means of evaluating success of the ESPP, *in-situ* bioaccumulation studies, where caged aquatic test species are temporarily placed in the Basin, are performed to measure the uptake of mercury and methylmercury. Asiatic clams (*Corbicula fluminea*) are used as the test organism.
- Analytical parameters are selected based on identification and quantification of COCs, site history, media affected (or potentially affected), and project objectives. Analytical methods selected for this project are obtained from acceptable sources such as the USEPA, the American Society for Testing and Materials (ASTM), Standard Methods (SM), and performance-based methods, consistent with federal and/or state regulations, and acceptable for generating data for comparison to risk-based screening levels.
- The sampling programs assume that general conditions are consistent throughout the sampling locations and that the analytical instrument response will be consistent for samples within the same medium. For example, surface water and sediment samples to be collected from locations within the OU-2 Basin will require the use of a boat, and ground water samples collected from monitoring wells will require low flow

sampling equipment. Because methylmercury analysis is not a standard method, the sampling program assumes an equal variance across samples from the same medium.

When collecting samples from the Basin and Round Pond during a flood event, ease of entry and exit is important for timely collection, processing, and sample packing as well as for health and safety reasons. The number of field and QC samples collected and the parameters analyzed for each of the current studies are presented in Table 2-1.

2.2 SAMPLING METHOD REQUIREMENTS

Project specific sampling methods are discussed in the WPs. Environmental information and samples will be collected in general accordance with the USEPA Region 4 EISOPQAM. Additionally, because the AWQC for the primary project COC, mercury, is so low (parts per trillion), a sampling method designed for trace metals collection is essential. Therefore, the surface water and groundwater samples are collected according to the low-level sampling procedures outlined in USEPA Method 1669. This sampling method allows for the collection of environmental samples while limiting low-levels of mercury from other sources. General environmental sampling protocols are discussed below. Preservations, holding times, container types, and required sample volumes for environmental chemistry parameters are shown in Table 1-4.

2.2.1 Low-Level Metals Sampling Procedures

Surface water and groundwater samples analyzed for low-level mercury by USEPA Method 1631E and methylmercury by USEPA Method 1630 Draft are collected using the "clean hands/dirty hands" sampling procedures specified in USEPA Method 1669.

A two-person team is required for sample collection. One member is designated as "Dirty Hands" and the other member is designated as the "Clean Hands." The "Dirty Hands" member is responsible for the preparation of the sampler (except the sample container itself), operation of any equipment, and other activities that do not involve contact with the sample. The "Clean Hands" member will handle operations which involve contact with the sample bottle and transfer of the sample from the collection device to the sample bottle.

Each member wears clean, lint-free outer clothing (such as Tyvek) and at least two pairs of non-talc gloves (wearing multiple layers of clean gloves will minimize disruption of sampling activities when

gloves are to be changed out). "Clean Hands" should wear an additional pair of shoulder-length polyethylene gloves. "Clean" sampling equipment and sample containers are obtained from the laboratory responsible for the testing. For OU-2 low-level mercury and methylmercury sampling, preservation and filtering is performed at the laboratory.

Sample equipment used for low-level mercury sampling is non-metallic or (when using pumps with some metal parts) the sample is not allowed to come in direct contact with metal parts in the equipment. Sample containers for mercury are made of fluoropolymer (FEP, PTFE, Teflon®) or glass because mercury vapors can diffuse in or out of other materials resulting in either contamination or biased-low results (Bloom, 1993).

Sample tubing is composed of fluoropolymer or styrene/ethylene/butylene/silicone (SEBS) material. When sampling from a boat, the boat and oars should be made of wood or fiberglass and cleaned with water from the sampling location. Gasoline- or diesel-fueled motors should be avoided. If motors are required then the engine should be shut off at a distance far enough from the sampling point to avoid contamination.

If mercury concentrations are known, samples are to be collected from lowest to highest concentrations. An effort should be made to collect samples in an upwind/upstream/upgradient location. A Field Blank is collected prior to collecting samples to monitor ambient mercury levels and an Equipment Blank is collected to verify that the equipment is free of contamination prior to the collection of a sample. Detailed low-level sampling procedures are presented in Appendix A.

2.2.2 Decontamination Procedures

The decontamination procedures for the equipment used to collect the samples, with the exception of samples for low-level mercury, are as follows:

- Rinse with ASTM Type II water
- Scrub with Liquinox™
- Rinse with ASTM Type II water
- Rinse with 5 percent nitric acid solution (Trace Grade)
- Rinse with ASTM Type II water
- Air dry

The decontamination procedures used for the equipment for collection of samples for low-level mercury are as follows:

- Liquinox™ scrub
- Rinse with tap water
- Double rinse with laboratory prepared reagent water

Dedicated or disposable equipment does not require decontamination. Large equipment, such as drilling augers, is steam cleaned with a Liquinox™ soap solution and double-rinsed with potable water.

2.2.3 Sample Containers

The laboratory provides the sample containers. The containers are commercially prepared by the vendor and are certified by production lot to be constituent free.

The laboratory assembles a sampling kit for each sampling event. The sampling kit includes the following items, when applicable: cooler(s), sample containers (with appropriate preservative), and Chain-of-Custody forms (Appendix B).

The laboratory provides rinsate blank containers containing the preservative for each type of sample collected. The same preservative is used for both blanks and samples.

Glass bottles are adequately protected from breakage by placing bottle in bubble bags, bubble wrap, or equivalent protection. Forms are placed in a waterproof bag. The cooler is then be sealed with packing tape and shipped to the laboratory. Both chemical and ecological samples are shipped via overnight delivery (*e.g.*, Federal Express) or hand carried to the laboratory.

2.2.4 Preservatives

Preservatives, when applicable, are supplied by adding the preservative to the sample containers. The preservatives used are American Chemical Society reagent grade or equivalent. Container and preservatives required for each method is presented in Table 1-4.

2.2.5 Sample Identification

Samples collected from OU-2 are labeled with the following information:

- Site/facility name
- Sampling location
- Names of sampling personnel
- Date and time of sample collection
- Unique sample identification number

Unique sample identification numbers are constructed as presented in task-specific WPs. An example Sample Label is presented in Appendix B.

2.3 SAMPLE HANDLING AND CUSTODY REQUIREMENTS

2.3.1 Field Custody Procedures

Field logbooks provide the means of recording data collection activities. As such, entries are described in enough detail so those individuals participating in the sampling can reconstruct a particular situation without reliance on memory.

Field logbooks are bound field survey books or notebooks. Logbooks are assigned to field personnel, but stored in the document control center when not in use. Each logbook is identified with the project-specific document number.

The logbook contains the following information for each activity:

- Location;
- Date and time;
- Individuals performing the activity; and
- Weather conditions.

Entries into the logbook contain a variety of information. At the beginning of each entry, the date, start time, weather, names of sampling team members present, and the signature of the person making the entry are entered. The names of visitors to the site, field sampling or investigation team personnel, and the purpose of their visit are also recorded in the field logbook.

Measurements made and samples collected are recorded. Entries are made in ink, signed, and dated and no erasures are made. If an incorrect entry is made, the information is crossed out with a single strike mark, which is initialed and dated by the sampler. Whenever a sample is collected or a measurement is made, a detailed description of the location of the station is recorded. The number of the photographs taken of the station, if any, is also noted. Equipment used to make measurements is identified along with the date of calibration.

Samples are collected in accordance with the sampling procedures documented in the WP. The equipment used to collect samples is noted, along with the time of sampling, sample description, depth at which the sample was collected, and volume and number of containers used. Sample identification numbers are assigned prior to sample collection. Field duplicate samples, which will receive a separate sample identification number, are noted under sample description. General field sampling responsibilities and protocols include the following:

- The field sampler is personally responsible for the care and custody of the samples until they are transferred or properly dispatched.
- Bottles are identified by use of sample labels with sample numbers, sampling locations, date/time of collection, and type of analysis.
- A properly completed chain-of-custody form (Appendix B) accompanies samples. The sample numbers and identifications are listed on the chain of custody form. When transferring the possession of samples, the individuals relinquishing and receiving signs, dates, and notes the time on the record. The chain-of-custody documents the transfer of custody of samples from the sampler to another person, to the laboratory, or to/from a secure storage area.
- Water, groundwater, sediment, and soil samples are properly packaged on wet ice at 4°C for all parameters with the following exceptions: sediment samples assayed for methylmercury and tissue samples are properly packaged on dry ice at 0°C for shipment and dispatched to the laboratory for analysis. A separate, signed custody record is enclosed in and secured to the inside top of each sample box or cooler. Shipping containers are secured with strapping tape and custody seals for shipment to the laboratory. The preferred procedure includes use of a custody seal attached to the front right and back left of the cooler. The custody seals are covered with clear plastic tape. The cooler is strapped shut with strapping tape in at least two locations.
- The chain-of-custody record identifying the contents accompanies all shipments. The original record and a second copy accompany the shipment. A third copy is retained by the sampler and placed in the project files.
- If the samples are sent by common carrier, a bill of lading should be used. Receipts of bills of lading are retained as part of the permanent documentation. If sent by

mail, the package is registered with return receipt requested. Commercial carriers are not required to sign off on the custody form as long as the custody forms are sealed inside the sample cooler and the custody seals remain intact.

- Samples are transported to the laboratory in sufficient time to insure that holding times are not exceeded prior to analysis.

2.3.2 Laboratory Custody Procedures

Laboratory custody procedures for sample receiving and login, sample storage and numbering, tracking during sample preparation and analysis, and storage of data is evaluated to document appropriate custody and integrity of the submitted samples. Laboratory custody procedures are presented in each of the laboratories' Quality Assurance Manual (QAM).

2.3.3 Final Evidence Files

The final evidence file includes but not limited to:

- Field logbooks;
- Field data and data deliverables;
- Photographs;
- Drawings;
- Soil boring logs;
- Monitoring well installation diagrams
- Laboratory data deliverables;
- Data validation reports;
- Data assessment reports;
- Progress reports, QA reports, interim project reports, etc.; and
- Custody documentation (labels, forms, air bills, etc.).

2.4 ANALYTICAL METHODS REQUIREMENTS

The following sections outline the analytical methods to be used for this project. These methods were obtained from the following sources:

- Test Methods for Evaluating Solid Waste, USEPA SW-846, 3rd Edition, and updates (USEPA, 1996b).

- "Methods for Chemical Analysis of Water and Wastes," EPA-600/4-79-020, March 1983, and subsequent revisions.

- Annual Book of ASTM Standards, (ASTM, 1987, 1998, 2007).

- Standard Methods for Examination of Water and Wastewaters, 18th, 19th, and 21st Eds. (American Public Health Association [APHA], et al., 1992, 1995, 2005) and subsequent editions.

- Bligh, E.G., and W. J. Dyer. 1959. A Rapid Method of Total Lipid Extraction and Purification. Canadian Journal of Biochemistry and Physiology. Vol 37 No. 8. pp. 911-917.

- Bloom 1989. Determination of Picogram Levels of Methylmercury by Aqueous Phase Ethylation, Followed by Cryogenic Gas Chromatography with Cold Vapor Atomic Fluorescence Detection. *Can. J. Fish. Aquat, Sci.*, Vol. 46, 1989.

- Allen, H.E., F. Gongmin, W. Boothman, D. DiToro, and J. Mahoney. 1991. Determination of Acid Volatile Sulfides (AVS) and Simultaneously Extracted Metals in Sediment. Draft Analytical Method for Determination of Acid Volatile Sulfide in Sediment.

Table 1-3 lists the analytical methods used for each sample matrix, along with standard laboratory QC criteria and method reporting limits. The methods are described below.

2.4.1 Field Analyses

The field analytical measurements to be utilized for a field project are summarized in Table 1-1. Surface water and groundwater samples are measured for some or all of the following field parameters:

Parameter	**Method**	**Equipment Make/Model**
pH (unit)	MCAWW 150.1	YSI Model 6920
Specific Conductance (mS/cm)	MCAWW 120.1	YSI Model 6920
Temperature (°C)	MCAWW 170.1	YSI Model 6920
Turbidity (NTUs)	MCAWW 180.1	YSI Model 6920
Redox Potential (mV)	SM 2580	YSI Model 6920
Dissolved Oxygen milligrams per liter (mg/L)	Air Saturation	YSI Model 6920
Pore-water pH (for sediment)	SW9045	YSI Model 6920
Pore-water ORP (mV) (for sediment)	ASTM D1498-76	YSI Model 6920

In-situ water quality data is collected prior to the collection of surface water, sediment, biota, and groundwater samples using a YSI Model 6920 or equivalent. The water quality meter is calibrated daily prior to use according to standard solutions. Calibration is checked in the middle and at the end of the sampling day. Calibration results are recorded in indelible ink on calibration logs (Appendix B) and/or field logbooks. Calibration procedures are presented in Appendix B.

2.4.2 Laboratory Analyses

Surface water, groundwater, sediment, soil, fish, and Asiatic clam samples are analyzed for the parameters presented in Table 1-1. These protocols are followed to allow for the generation of representative data. The standard operating procedures (SOPs) for methods to be performed by the laboratories are incorporated herein by reference and a general description of each method is presented below. Additional test methods required for future studies will be presented in the task specific WP, if different from or in addition to those presented herein.

2.4.2.1 HEXACHLOROBENZENE BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

Hexachlorobenzene is one of three COCs (mercury, hexachlorobenzene, and DDTR) specific to OU-2 and is analyzed using USEPA Method 8270C. Method SW8270C was selected to aid in determining the presence or absence of hexachlorobenzene contamination identified in previous studies. The samples are extracted using USEPA Methods 3520C (continuous liquid-liquid extraction) for waters and 3546 (microwave extraction) for sediments/soils. In Method 3520C, the water sample is placed into a continuous liquid-liquid extractor, adjusted, if necessary, to a specific pH, and extracted with methylene chloride for 18 to 24 hours. In Method 3546, a 30-gram aliquot of soil/sediment is mixed with anhydrous sodium sulfate and solvent extracted in an enclosed vessel under heat and pressure using microwave extraction. The soil extract is separated from the sample by vacuum filtration. The extract for both liquids and soils is then dried with sodium sulfate, and concentrated. This sample extract is directly injected into a gas chromatograph (GC) in which the SVOCs are qualitatively separated and quantitatively detected with a mass spectrometer.

2.4.2.2 DDTR and Hexachlorobenzene by GC with Second Column Confirmation

DDTR is analyzed by USEPA Method 8081A. Hexachlorobenzene in tissue is also analyzed by 8081A to allow for quantitation of all analytes using a single extract. DDTR includes the 4,4' and 2,4' isomers of DDD, DDE, and DDT.

The samples are extracted using USEPA Methods 3520C (continuous liquid-liquid extraction) for waters, 3546 (microwave extraction) for sediments/soils, and 3540C for clams/fish. In Method 3520C, the water sample is placed into a continuous liquid-liquid extractor, adjusted, if necessary, to a specific pH, and extracted with methylene chloride for 18 to 24 hours. In Method 3546, a 30-gram aliquot of soil/sediment

is mixed with anhydrous sodium sulfate and solvent extracted in an enclosed vessel under heat and pressure using microwave extraction. The soil extract is separated from the sample by vacuum filtration. In Method 3540C, homogenized tissue is mixed with sodium sulfate and extracted with methylene chloride. The extracts from the various matrices may require cleanup by appropriate methods such as gel permeation chromatography (GPC) or Florisil cartridge cleanup. The sample extracts are injected into a GC using the solvent flush technique and split between two dissimilar columns for simultaneous primary and confirmation analysis. The compounds in the split GC effluent are detected by electron capture detectors (ECD).

2.4.2.3 Mercury Cold -Vapor Atomic Absorption

Total and dissolved mercury in water, total mercury in soil and sediment, total mercury in clams and fish, and associated QC samples are analyzed by USEPA Methods 7470A, 7471A, and 245.6, respectively. Mercury is the primary COC for OU-2.

Prior to analysis, samples must be digested using the method specific digestion procedures. In Method 7470A, samples are acidified, digested with potassium permanganate and potassium persulfate, and the excess permanganate removed with sodium chloride/hydroxylamine hydrochloride solution. In Method 7471, soil and sediment samples are acidified and digested with potassium permanganate, and the excess permanganate removed by addition of sodium chloride/hydroxylamine hydrochloride solution. In Method 245.6, tissue is dissolved using sulfuric and nitric acids, and digested with potassium permanganate and potassium persulfate. Hydroxylamine is added to remove excess permanganate. Extracts from each matrix are diluted to volume with reagent water.

Mercury analysis, a cold-vapor atomic absorption method, is based on the absorption of radiation at the 253.7-nm wavelength by mercury vapor. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes though a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance (peak height) is measured as a function of mercury concentration using a linear calibration curve.

2.4.2.4 Methylmercury Cold-Vapor Atomic Fluorescence

Methylmercury in water, soil/sediment, and fish/clams and associated QC samples is analyzed by the procedure published by Bloom (1989), derived from USEPA Method 1630 (draft). The method is a cold

vapor atomic fluorescence technique, is based on the emission of 254 nm radiation by excited mercury (Hg^{2+}) atoms in an inert gas stream. Tissue samples are digested with potassium hydroxide/methanol reagent prior to analysis. Methylmercury in water and soil/sediment samples is distilled or extracted into a clean water matrix. An ethylating agent is added to the digestate/distillate/extract to form a volatile methyl-ethylmercury derivative. The sample is then purged onto a graphite carbon trap as a means of preconcentration of interference removal. The sample is then isothermally chromatographed, pyrolitically broken down to elemental mercury, and detected using a cold vapor atomic fluorescence detector.

2.4.2.5 Low-level Mercury Cold-Vapor Atomic Fluorescence

Low-level mercury in water and associated QC samples are analyzed by USEPA Method 1631E, which can detect mercury at concentrations as low as 2 ng/L as Hg. This method was selected to determine mercury, the primary COC at a level that is below the AWQC.

Method 1631E, a cold vapor atomic fluorescence method, is based on the emission of 254 nm radiation by excited mercury (Hg^{2+}) atoms in an inert gas stream. Mercuric ions in the oxidized sample are reduced to Hg^{2+} with stannous chloride ($SnCl_2$) and then purged onto gold-coated sand traps as a means of preconcentration and interference removal. Mercury vapor is thermally desorbed to a second analytical gold trap, and from that into the fluorescence cell. The fluorescence (peak area) is proportional to the quantity of mercury collected, and is quantified using a standard curve as a function of the quantity of sample purged.

2.4.2.6 Pore-water Mercury and Methylmercury

Analysis for mercury and methylmercury contained in the pore water of the soil/sediment samples are performed as described above following preparation using pressurized filtration or vacuum filtration.

Soils and sediments are filtered under pressure of an inert gas or vacuum filtered using an acid rinsed 0.45 micron Teflon filter.

2.4.2.7 Iron, Manganese, Molybdenum, and Selenium by ICP

Metals in water and soil/sediment and associated QC samples are analyzed by USEPA Methods 6010B. Prior to analysis, water samples are digested with nitric acid, refluxed with hydrochloric acid, and brought

up to volume for analysis as described in Method 3010A. Soil/sediment samples are digested using nitric acid and hydrogen peroxide, refluxed with hydrochloric acid, filtered, and refluxed again as described in Method 3050B.

The metals analysis involves multi-elemental determinations by inductively coupled plasma-atomic emission spectroscopy (ICP-AES) using simultaneous and/or sequential instrumentation. The instrument measures characteristic atomic-line emission spectra by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element specific spectra are produced by radio frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the line spectra are monitored at specific wavelengths by a photosensitive device. A background correction technique is required to compensate for the variable background contribution to the determination of the analytes. Analytes are quantitated using a linear calibration curve. .

2.4.2.8 Total and Dissolved Organic Carbon (TOC)

TOC in soil/sediment, and TOC and dissolved organic carbon (DOC) in water samples will be determined by Standard Methods 5310B or USEPA SW-846 Method 9060. Organic carbon in the samples is converted to carbon dioxide by catalytic combustion. The carbon dioxide is then measured directly by an infrared detector or converted to methane and measured by an FID.

2.4.2.9 Sulfate

Sulfate in water samples will be determined by USEPA Method SW9038. In Method 9038 sulfate is converted to a barium sulfate suspension and the resulting turbidity is measured by a filter photometer at 405 nanometers (nm). A 100-milliliter sample aliquot is required for this test.

2.4.2.10 Sulfide

Sulfide in water samples will be determined by USEPA Method SW9030. In Method 9030 acid-soluble sulfide is separated from the matrix by the addition of sulfuric acid, heated, and the resulting hydrogen sulfide is distilled through a zinc acetate gas to form a zinc sulfide precipitate. The precipitate is quantitated titrimetrically or determined colormetrically.

2.4.2.11 Acid Volatile Sulfide/Simultaneously Extractable Metals

Acid volatile sulfide (AVS) and simultaneously extractable metals (SEM) in soil/sediment are analyzed by the method in Allen et al. (1991). Sulfide in the sample is converted to hydrogen sulfide by the addition of hydrochloric acid at room temperature. The hydrogen sulfide is then purged from the sample by an inert gas and trapped in a sodium hydroxide solution. Addition of a mixed diamine reagent (MDR) causes the sulfide to convert to methylene blue which is then measured on a spectrometer. The concentration of metals associated with the sulfide is then determined by analyzing the purged sample for metals by ICP-MS (cadmium, copper, nickel, lead, silver, and zinc) and for mercury by Cold Vapor Atomic Fluorescence as previously described.

2.4.2.12 Miscellaneous Test Methods

Miscellaneous test methods for water include total alkalinity, hardness, total dissolved solids (TDS), and TSS. Miscellaneous test methods for soil/sediment include pH, oxidation-reduction potential (ORP), percent moisture, density, and grain size. Miscellaneous test methods for clams/fish include percent moisture and percent lipids.

2.5 QUALITY CONTROL REQUIREMENTS

A qualified engineer or scientist reviews documents involving engineering or scientific evaluation, interpretation, or judgment. A qualified engineer or scientist is one who has suitable experience with the techniques employed, conditions evaluated, and technologies involved and is authorized by corporate policy to practice in the discipline covered.

The quality control procedures specified in the current SW-846 methodologies and specified USEPA methods are followed in the laboratory and the field.

2.5.1 Field Quality Control

Field sampling procedures call for preparation and submittal of the following types of QC samples.

- Rinsate blanks - are prepared in the field to demonstrate that a sampling device (*e.g.*, auger) has been effectively cleaned. The sampling device is filled with organic-free, deionized water that will then be poured through the device, transferred to the

appropriate sample bottles, preserved, and returned to the laboratory for analysis. One rinsate blank is collected per non-dedicated sampling tool per media used.

- Field (blind) duplicates - Two sets of samples from a single source are prepared, labeled with unique sample numbers, and submitted to the laboratory without cross-referencing data and without identification as duplicates on the parameter request sheet. One blind duplicate is collected for every 10 environmental samples collected for each matrix type.

- Field blank – A water blank, using water provided by the laboratory, is prepared in the field once per sampling day for standard analyses. An additional field blank is collected if weather or site conditions notably change during a day of sampling. Two field blanks are collected during low-level sampling; one in the morning and one in the afternoon.

- Split Samples – A single sample divided into two equal parts for analysis or two samples collected independently at a sampling location during a single act of sampling. One sample (the QA split) is then sent to a USEPA-designated laboratory for independent verification of the results for that location. QA split samples will be collected upon request by the USEPA.

- Performance Evaluation Samples – Fortified samples prepared by an independent vendor and sent to the field and labeled as a field sample or sent directly to the laboratory for analysis. Sample aliquots are fortified (spiked) with known amounts of site-specific COCs. Performance evaluation samples monitor the laboratory's performance. Performance evaluation samples will be prepared and analyzed upon request by the USEPA.

2.5.2 Laboratory Quality Control

To obtain data on the precision, accuracy, and recovery, the analytical laboratory analyzes the QC samples as specified in Section 1.7.2 and Tables 2-2 and 2-3. The control limits and corrective actions for each parameter are specified in each laboratory analytical method SOP.

For inorganic analyses of soil and water, the analytical methods require analyses of the following QC samples.

- Calibration verification following instrument calibration and once every tenth sample thereafter through the working day.

- Laboratory blank verification at instrument calibration and once every tenth sample thereafter through the working day to check instrument drift.

- Method blank analysis at a rate of one per batch of samples or one per 20 samples of a single matrix, whichever is more frequent, to determine contamination levels during preparation.

- MS/MSD analyses at a rate of one per batch of samples for each matrix type (*e.g.*, soil, water) and concentration level (*e.g.*, low, medium) or one in 20 samples, whichever is more frequent. The MS/MSDs are used to check for the ability to accurately and precisely recover compounds of interest from the matrix.

The results of analyses of these QC samples will be used as independent, external checks on laboratory and field contamination.

2.5.3 Records

Records of samples maintained by the laboratory include the following:

- Sample receiving logbook - to log the samples when they are received and assigned a batch number.
- Standards logbook - to record the preparation of standards in the laboratory.
- Instrument logbook - to record the samples analyzed and QC.
- QC logbook - to record day-to-day QC data obtained from the analysis of a batch. QC summary sheets are used as a convenient method to file batch QC information by parameter.
- Central file - to store the record of the raw data and final data for every batch.
- QC charts used to track performance on individual analyses and instruments and to give early indication of analyses that may be going out of control.

Records of samples maintained by Olin and/or Olin's subcontractors include the following:

- Field log book – to record the time, date, place, person(s), and parameters collected on the sample, the conditions at the time of sampling, any problems encountered, and corrective actions taken during sampling (if required)
- Calibration forms – to record the calibrations and standards used to calibrate field equipment
- Chain-of-Custody - to record the time, date, and parameters to be analyzed on the sample
- Analytical data – to present the chemical/physical data
- Electronic Data Deliverable (EDD) – to store the chemical/physical data of the sample into a database from which the data can be manipulated into tabular reports, queried, and organized

- Plans – to present the why, the type, the how, and the what the sample is to be collected for – present the project DQOs
- Reports – to present the chemical/physical data of the sample(s) to the client. Reports can also include any outputs from a database, modeling results, calculations, figures, etc.
- Project records are converted to electronic media and stored in a project database management system.

2.6 INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE

The preventive maintenance procedures for field and laboratory equipment are presented below.

2.6.1 Field Equipment

Field monitoring and analytical equipment are maintained in accordance with the manufacturers' recommended schedules and procedures. Maintenance activities are documented by either field or laboratory personnel. Calibration is performed on a routine basis and as otherwise required. Calibrating equipment is routinely recalibrated and documented. Routine inspection of equipment is intended to identify problems requiring maintenance before they cause a major disruption of the field monitoring or analytical activities or adversely affect the validity and precision of the data being measured.

2.6.2 Laboratory Equipment

The laboratory usually maintains a service contract with the laboratory equipment manufacturers for major instrumentation in order to minimize downtime of the analytical systems. A service engineer performs necessary preventive maintenance. In the event that analytical equipment used in this study is unable to perform the necessary analyses, appropriate secondary equipment owned by the laboratory is reconfigured and dedicated to complete the scheduled analytical laboratory work. A supply of spare parts will be maintained to minimize downtime.

Each analyst is responsible for conducting a daily inspection of critical systems on instruments under their charge. Inspections include vacuum lines and pumps for GC/MS, automatic injection systems, controlled reagent-feed motors, temperature-controlled ovens in GCs, capillary columns, detectors and support systems, gas control system for Atomic Absorption (AA)'s, and many others. Wear-dependent items such as septa on GC injection systems are to be replaced as needed. The performance of instruments is to be

checked against known standards at the beginning of each working day or shift. Failure to achieve proper performance indicates a system problem, which will be resolved by laboratory personnel or by the manufacturer's service representative.

In addition, laboratory personnel or the manufacturer's service representative service the working systems according to a schedule. A record of service and repairs, whether accomplished by laboratory personnel or by the manufacturer's service representative, is maintained in a logbook kept with each instrument. Table 2-4 presents the general preventative maintenance of laboratory equipment.

2.7 INSTRUMENT CALIBRATION AND FREQUENCY

The following sections discuss calibration procedures and frequency. Availability and types of standards are also discussed.

2.7.1 Field Instrument Calibration

Instruments and equipment used to gather, generate, or measure environmental data are calibrated with sufficient frequency and in such a manner that accuracy and reproducibility of results are consistent with the manufacturer's specifications. Table 2-5 presents the typical field calibration frequency and corrective action procedures.

Equipment to be used during the field sampling is examined to determine its operating condition. This includes review of the maintenance requirements for each instrument. Equipment calibration results from previous equipment use may be reviewed.

Surface water and groundwater are measured for several field parameters including pH, temperature, specific conductivity, dissolved oxygen, oxidation-reduction potential, turbidity, and salinity. The meter or meters are calibrated according to manufacturer's specifications at the beginning of each day of use. Calibrations are verified at the middle of each day. The manufacturer specifies the calibration procedures for the instrument. Calibration results are documented by including the following:

- Date calibrated;
- Person who calibrated the instrument;
- The instrument number (serial number or other identification number);
- The results of the calibration; and

- Identification of the calibration standard (source, type, concentration).

Field logbooks are used to record calibration dates, results, statistics, and the resulting data measurements. These logbooks include maintenance and repair reports. Entries are signed and dated by the personnel performing the required action.

2.7.2 Laboratory Calibration Standards

Calibration standards are traceable to the National Institute for Standards and Technology (NIST) or American Association for Laboratory Accreditation (A^2LA), whenever such standards are available. Commercial sources of standards and reagents are to be checked for purity, and approved prior to their use in analysis.

Standards prepared for use throughout the laboratory are uniquely identified and entered in a bound standard notebook with information regarding the preparation of that standard (*i.e.*, date, technician, name of each compound and amount used, final volume, and solvent used). Standard containers are labeled so that the standard is traceable to the standard's identification, lot number, manufacturer, and date.

The instrument response obtained for each compound in a newly prepared standard is compared to the response obtained from the previous standard. The two standards must pass calibration verification criteria (for all but a few compounds recognized as being chromatographically atypical) before the new standard may be used. The new standard may not be used until the discrepancy has been resolved. The working lifetime of standard preparations is dependent upon the compound types comprising the standards.

2.7.3 Chemical Analysis Calibration

Instruments are calibrated before being put into service and will be recalibrated at regularly specified intervals consistent with the manufacturer's recommendations. Instrument response is subjected to checks between the regular recalibrations. The nature and frequency of such checks are dictated by the standard operating procedures practiced by the analytical laboratory. The analytical laboratory maintains adequate records of calibrations, recalibrations, and in-service checks of instruments. The schedule of checks depends on the experience of the laboratory's maintenance needs. Calibrations are traceable to primary

standards of measurement. Where the concept of traceability of measurements to primary standards is not applicable, the laboratory provides satisfactory evidence of correlation or accuracy of test results.

Analysts, assistant managers, lab managers, and QA staff inspect calibration data for completeness and validity. Forms are checked for arithmetic and procedural errors. Recurring errors, either caused by individual operators or by ambiguously worded instructions, are brought to the attention of the department senior laboratory staff or laboratory management for corrective action. Calibrations will meet criteria as specified by the applicable methods.

2.8 INSPECTION/ACCEPTANCE REQUIREMENTS FOR SUPPLIES

Two techniques are utilized to document that supplies used for analysis are of acceptable quality. The first technique involves the supply vendor stockpiling a specific lot number of a consumable product (such as solvents or reagents) for use by the laboratory. The quality of a reagent from one production lot is usually consistent. The second technique involves quality verification of newly obtained supplies by analysis of blanks and/or control samples to verify consistency between the new and old supply of material. The laboratory is proactive in verifying the quality of new reagents prior to the consumption of the existing supply to facilitate an acceptable transition to the new supply. Lists of major supplies will be presented in each individual WP.

2.9 DATA ACQUISITION REQUIREMENTS (NON-DIRECT MEASUREMENTS)

Data used for project decisions and reports that were obtained from data tables, other sources, or calculations are verified by at least two project personnel prior to use. Data tables or other data summaries include “Prepared by” and “Checked by” fields at the end of the data to document this two-person review process. The data obtained from non-direct measurement sources may also include: geological, hydrological, and meteorological data including demographics, land use, and endangered species. Published literature regarding the physiography, climate, geology, and hydrology in Alabama was reviewed and the historical databases compiled by the U.S. Geological Survey (USGS) and data from a number of consulting engineering firms and contractors were also reviewed to further assess geochemical conditions and constituent distribution in the media to develop the conceptual site model. In addition, a literature review was conducted for the mercury methylation report (MACTEC, 2008) which included reliable sources such as the USEPA. Literature cited is presented in the “References” section of the document.

2.10 DATA MANAGEMENT

Data resulting from laboratory analysis are consistent with the appropriate methods and equations stated in the procedure. Individual laboratory supervisors review data before forwarding it to the data management supervisor. The laboratory QA Manager reviews final reports for error or deviations before release. Final reports include the Quality Control Summary data required to perform data assessment. Procedures used for analyses are compared with the reference methods. Discrepancies or deviations are be noted and explained.

The data generated during the sample collection and analysis are centralized into one project file including information about the instrument conditions. The data management system allows review by project personnel. Personnel must be approved to access or save to the project file.

The laboratory may submit an EDD in the EQuIS™ format where applicable. The data manager uploads the data into a temporary database where the EDD undergoes a review process. Errors that cannot be resolved by Olin or Olin's subcontractors are communicated to the laboratory. The laboratory may be required to submit a corrected EDD. Once an EDD is processed without errors, the data manager uploads the data into a permanent database specific to the project. Outputs from the database are checked for accuracy by another person other than the person who produced the output. Both persons initial and date the output deliverable. The data manager maintains a record of any data transactions and an electronic copy of all outputs. Only the data manager has access to the permanent database and any changes and or edits can be performed by the data manager. Edits to the database require documentation. If the data are to be electronically uploaded to a USEPA database, the data manager and PM meet with the EPA representative(s) to discuss the format required to upload the data to ensure it meets the Office of Information Resource Management requirements specific to the USEPA Region.

3.0 ASSESSMENTS/OVERSIGHT

3.1 REQUIRED ASSESSMENTS AND RESPONSE ACTIONS

Assessments and/or oversight of the laboratory and field activities, project status, and data quality may be performed prior to, during, and after project activities. A Management System Review (MSR) is performed on each laboratory prior to project initiation. The QA office or designated personnel will perform the annual laboratory MSRs. Technical System Audits (TSA) of field activities are being coordinated and performed by EPA Region IV. Project status assessments are performed monthly by the Project Manager and documented in the Monthly Progress Reports. Data Quality Assessments (DSAs) are performed on the lab and field data via validation by project chemists. Detailed data verification and validation activities are discussed in Section 4.0 Senior professionals perform the audit/assessment, document performance, and initial corrective action if necessary. Reports generated from the assessments and/or oversight activities are presented in Section 3.2.

3.2 REPORTS TO MANAGEMENT

A summary of QA/QC related reports are listed below:

- Data Validation Report
    - Provides a summary of the quality of the data and a description of qualifiers applied to the data. Section 4.1.2.3 provides a detailed description of a validation report. The Data Validation Report is submitted to the project PM.
- Data Assessment Reports
    - Presents the sample data to the client as it relates to the project DQOs. The Data Assessment Report (will be named for the specific plan it supports, *i.e.* ESPP Report, Treatability Report, etc.) is submitted to Olin and Olin's subcontractor PMs with subsequent submittal to the regulatory agencies.
- Nonconformance Reports (if any)
    - Olin requires subcontractors to provide documentation of a nonconformance. The nonconformance report provides a description, cause, potential harm, and the corrective action taken for a nonconformance. Figure 3-1 presents a Nonconformance and Corrective Action Report Form.
- Monthly Progress Reports

- Summarizes project activities and tasks conducted. Monthly Progress Reports are in compliance with Section VII. E. of the Consent Order (USEPA, 1990). The Monthly Progress Reports include:

    1) A description of the actions taken toward achieving compliance with the Consent Order;
    2) Results of sampling and testing and other data;
    3) Plans and procedures completed;
    4) Description of actions, and plans which are scheduled for the next month;
    5) Information regarding percent completion and any unresolved delays that may affect the future schedule and a description of the efforts to mitigate the delays.

The Monthly Reports are submitted to the USEPA and ADEM on the tenth day of each month.

4.0 DATA VALIDATION AND USABILITY

4.1 DATA REDUCTION, VALIDATION AND REPORTING

Proper data management is as important as proper analysis and custody procedures in ensuring representativeness. Data reduction, validation, and reporting procedures function to control data handling from field collection through laboratory analysis and data processing to the point where data are turned over to the data user.

Data quality and utility depend on many factors, including sampling methods, sample preparation, analytical methods, QC, and documentation. Subcontractors, such as laboratories or sampling personnel, must be advised of applicable documentation and procedural requirements. Once the data are assembled, satisfaction of validation criteria will be documented as listed below. Chemical data must meet criteria of: (1) quantitative statistical significance; (2) custody and document control; and (3) sample representativeness.

To determine the quantitative statistical significance of chemical data, items will be documented as appropriate (*e.g.*, with laboratory records, with laboratory SOPs by reference to an approved SOP manual, or with equipment manufacturer/supplier records). The laboratory accomplishes this through the tracking of method QC with control charts. Data tracked via control charts are continually updated and produce the statistical ranges for analyte- and method-specific precision, accuracy, and detection limits. These limits are in turn used by the data validator to assess the project data.

Documentation may be either direct (for example, listing of dates, names, and methodologies) or by reference to existing documents. Referenced documents are specifically identified. The precise and retrievable location of nonstandard documents (*e.g.*, in-house procedure manuals, chain-of-custody forms, and laboratory reports) is stated.

To determine sample representativeness, the following items must be checked:

- Compatibility between field and laboratory measurements or suitable explanation of a discrepancy;
- Sample preservation technique and holding time;

- Sample storage within suitable temperature, light, and moisture conditions;
- Use of proper sample containers;
- Use of proper sample collection equipment;
- Use of proper decontamination procedures;
- Use of proper laboratory preparation techniques; and
- Proper sample location selection.

To evaluate the physical data that support the analytical data, the following items will be documented:

- Sampling date and time;
- Sampling team noting the observation taker, recorder, and team leader;
- Sampling location and physical description sample depth increment for soils;
- Sample collection techniques;
- Field preparation techniques (*e.g.*, compositing);
- Visual classification of sample using an accepted classification system;
- A description of the methodology used, and a rational for the use of that methodology (as included in the project WP); and
- Examination of documentation of record keeping practices.

4.1.1 Field Measurements

Raw data from field measurements and sample collection activities are appropriately recorded in the field logbook. Field logbooks will be reviewed and checked by a second project team member. If the data are used in the project reports, they are reduced or summarized and the method of reduction is documented in the report.

4.1.2 Laboratory Analysis

The following sections describe the data reduction, validation, and reporting procedures to be performed by the laboratory, Olin, or Olin's subcontractors.

4.1.2.1 Data Reduction

The analyst performs the analysis and enters the data on the parameter bench sheet and corresponding data station(s). Bench sheets contain necessary information to establish sample identity, integrity, calibration evaluation, and analytical observations and results. A bench sheet key is provided to the analyst who specifies the way in which bench are sheets to be filled out (*i.e.*, notation, significant figures, etc.), the data reduction formula, and the QC samples required and their control criteria. QC samples include duplicates, MS or MSDs, continuing calibration verification samples (CCVs), etc. The use of rounding rules and significant digits for numerical data are in accordance with EPA-600/4-79-019 publication, "Handbook for Analytical Quality Control in Water and Wastewater Laboratories."

The laboratory for the duration of the study will keep raw, preliminary, and final data and instrument readouts (*e.g.*, chromatograms, printed digital readouts, etc.). Ultimately, data is archived along with other project records.

4.1.2.2 Data Reporting and Validation

Laboratory – Data can be reported by the laboratory as both hardcopy and EDD. The hardcopy report contains the elements described previously in Section 1.9. Prior to reporting, the data are validated by laboratory supervisors. Final review of the data is performed by the QA manager and/or PM. Hand calculations and manipulations are checked and verified. The EDD is produced per the format requested and checked via the laboratory LIMS. Standard turn-around-times for hardcopy data deliverables is 30 calendar days and the EDD is 45 calendar days from sample receipt unless otherwise noted.

Olin/Olin's subcontractors - Data are summarized as they are generated and submitted to the project team. The data are considered preliminary until completion of review and validation.

One hundred percent of the data is validated prior to use. A full data validation by Olin's or Olin's subcontractor project chemist, who is an individual separate from the sampling team, is conducted for data used in risk assessments. Partial validation is used for other data that are not used in risk assessments and may be reviewed by staff personnel under the supervision of the project chemist. Data validation is performed using criteria described in this QAPP and specific analytical methods.

The data review and validation consists of checking samples and QC results to demonstrate that the analyses are within prescribed criteria for precision, accuracy, completeness, sensitivity, blank contamination, etc. In addition to tabulated results, instrument readouts (*e.g.*, calibration curves, summary reports, etc.) are checked.

The partial review consists of an evaluation of the routine QA/QC performed by the laboratory. This includes review of the following QA/QC controls:

- Sample preservation;
- Holding times;
- Extraction/preparation/method blanks;
- Laboratory control samples;
- Matrix spike and matrix spike duplicates;
- Surrogate spikes, if applicable;
- Trip blanks;
- Field blanks;
- Equipment rinsate blanks; and
- Field duplicates.

A full validation included the QA/QC elements reviewed in the partial plus the following elements:

- Initial and continuing calibration;
- Calibration verification samples;
- Tuning criteria;
- Internal standards;
- Serial dilutions and post digestion spikes;
- Breakdown and second column confirmation; and
- Raw data printouts and calculations.

If data points are qualified, they receive data qualifiers. The qualifiers indicate if results are usable as-is, usable as-estimated or unusable (rejected). A case narrative id generated for each analytical package submitted by the laboratory. This narrative represents a summary on the quality of the data. A Data Validation Checklist is presented in Appendix C. Standard data qualifiers are used to classify data as to their conformance to QA/QC requirements. The data qualifiers used in this project are described in Table 4-1.

Validation of data obtained from field measurements is also performed. Data validity is evaluated by checking calibration procedures utilized in the field as appropriate and by comparing the data to previous

measurements obtained, if available. Variations in data that cannot be explained are assigned a lower level of validity and are used for limited purposes.

4.2 CORRECTIVE ACTIONS

Corrective action is initiated upon identification of problems either through systems or by standard QC data review. Essential steps in the corrective action system are:

- Identifying and defining the problem;
- Assigning responsibility for investigating the problem;
- Investigating and determining the cause of the problem;
- Determining a corrective action to eliminate the problem;
- Assigning and accepting responsibility for implementing the corrective action;
- Implementing corrective action and evaluating its effectiveness; and
- Verifying that the corrective action has eliminated the problem.

The laboratory QA Officer, Olin, or Olin's subcontractor may issue a Stop Work Order if appropriate corrective actions are not taken and the non-conformance is considered significant. Prior to issuing a Stop Work Order, the QA Officer, Olin, and/or Olin's subcontractor attempt to resolve outstanding non-conformances. A Nonconformance and Corrective Action Form (Figure 3-1) is generated to document a project non-conformance. Refer to Section 4.2.2 for detailed laboratory corrective action procedures.

4.2.1 Field Corrective Action

Project members who know or suspect that an activity is not being performed in accordance with those requirements must identify project tasks or items that do not conform to the QA/QC requirements based on field procedures. The Olin PM is informed of such defects and act in a timely manner to verify if corrective action is necessary.

If errors in field procedures are found during the observation and/or review of field activities, corrective action is initiated. The protocols outlined to correct the nonconforming activity are used to meet specified QA/QC requirements. The activity is reviewed by Olin or Olin's subcontractor to identify the source of the problem and develop a plan to correct the nonconforming items. Corrective actions for field sampling and testing problems are developed with assistance from the field team as follows:

- No additional work dependent on the nonconforming activity is performed until the problem is corrected; and

- Olin or Olin's subcontractor is notified when corrective actions are complete and then perform follow-up audits to confirm the resolution of the nonconformance.

If the problem(s) has been corrected to the satisfaction of Olin's PM, the activity may resume. Table 2-5 presents a summary of the field corrective actions.

4.2.2 Laboratory Corrective Action

The need for laboratory corrective action originates when an inadequacy is found in the method of analysis (*e.g.*, inappropriate calibration) or a determinate error occurs (*e.g.*, calibration error due to standards failure). Failures of the first kind are precluded by the laboratory and regulator/client audits that evaluate analytical SOPs. The analytical SOPs incorporate mechanisms to detect the existence of determinate errors and specify the procedures to correct them. Depending on the nature of the corrective action, it is classified as one of two types, immediate or long-term. Immediate corrective actions are the correction of procedures or the repair of instrumentation that is working improperly. Long-term corrective actions eliminate analytical problems by correcting systematic errors.

4.2.2.1 Response

Many times the source of a nonsystematic problem is obvious to the analyst and can be corrected immediately. Immediate corrective action routinely made by laboratory analysts should be documented as normal operating procedures in instrument logbooks. The supervisor and analyst should compile a list of commonly encountered problems and the appropriate routine corrective actions (in addition to manufacturer's troubleshooting guides). The operations manager and QA manager are responsible for approving corrective actions.

For calibration failures, corrective action consists of analyzing standards to establish a new initial calibration or continuing calibration. In cases where MS/MSD criteria fail but LCS criteria are within control limits, corrective action consists of qualification of the sample. If internal standards or surrogates fail criteria, the sample is re-extracted or reanalyzed and both analyses are reported. Method/prep blanks and associated samples are re-extracted and analyzed if concentrations greater than the RL are detected in the blank sample with the exception of common laboratory contaminants (*i.e.*, 2-butanone) which must not exceed three times the RL or the associated samples do not contain the constituent or contain it greater than five times the blank amount. A LCS sample with failures of the target analytes is re-extracted and

reanalyzed along with its associated samples. Data qualification flags are added to those analytes affected by out of control QC data or non-conformance to sampling, handling, or shipping requirements.

4.2.2.2 Reestablishment of Control

Corrective action is not complete until the problem has been permanently solved. Follow-up action to ensure that the problem remains corrected is a vitally important step in the corrective action procedure. Once a problem has been technically defined, the operations manager and the QA manager discuss the problem and jointly take the following steps:

- Determine that specific corrective action is needed to eliminate the problem and assign responsibility for investigating, implementing, and documenting the situation.
- Set a time schedule for determining the required action;
- Assign responsibility and time schedule to implement the desired action;
- Establish desired effectiveness of the corrective action and implement the correction; and
- Verify that the corrective action has eliminated the problem and document the incident for review and lessons learned.

4.2.2.3 Documentation

To provide a complete record of QC activities, QC issues and corrective actions applied must be documented. Historical records assist laboratory management in identifying long-term corrective actions, such as personnel training, replacement of instrumentation, and improvement of sampling procedure. A corrective action requires defined responsibilities for scheduling, performing, documenting, and assuring the effectiveness of the action. A Corrective Action Tracking Log form or Quality Problem Report is used to document the above steps and aid in written communication between the analyst and laboratory management.

5.0 REFERENCES

Allen, H.E., F. Gongmin, W. Boothman, D. DiToro, and J. Mahoney. 1991. Determination of Acid Volatile Sulfides (AVS) and Simultaneously Extracted Metals in Sediment. Draft Analytical Method for Determination of Acid Volatile Sulfide in Sediment.

ASTM, 1987, 1998, 2007. Annual Book of ASTM Standards.

Environment Australia, 2002. National ocean disposal guidelines for dredged material. May 2002. http://www.environment.gov.au/coasts/pollution/dumping/guidelines/investigation10.html

Benoit, J., 1999. The effect of sulfate and sulfide on mercury methylation in Florida Everglades sediments. EPA Grant Number U915152. http://es.epa.gov/ncer/fellow/progress/97/benoitja99.html

Bligh, E.G., and W. J. Dyer. 1959. A Rapid Method of Total Lipid Extraction and Purification.

Canadian Journal of Biochemistry and Physiology. Vol 37 No. 8. pp. 911-917.

Bloom 1989. Determination of Picogram Levels of Methylmercury by Aqueous Phase Ethylation, Followed by Cryogenic Gas Chromatography with Cold Vapor Atomic Fluorescence Detection. *Can. J. Fish. Aquat, Sci.*, Vol. 46, 1989.

Compeau, G.C., and R. Bartha, 1985. *Sulfate-reducing bacteria: principal methylators of mercury in anoxic estuarine sediment.* Applied and Environmental Microbiology 50:498-5020.

Fleming, E.J., E.E. Mack, P.G. Green, and D.C. Nelson. 2006. M*ercury Methylation from Unexpected Sources: Molybdate-Inhibited Freshwater Sediments and Iron-Reducing Bacterium.* Applied and Environmental Microbiology 72:457-464.

Fleming, E. J. and D C. Nelson, 2006. Contribution of iron-reducing bacteria to mercury methylation in marine sediments. *Coastal Environmental Quality Initiative.* Paper 040. December 8, 2006. http://repositories.cdlib.org/ucmarine/ceqi/040

Gilmour, C.C., E.A. Henry, and R. Mitchell. 1992. *Sulfate stimulation of mercury methylation in freshwater sediments.* Environ. Sci. Technol. 26:2281-2287.

Gobeil, C. and D. Cossa, 1993. Mercury in sediments and sediment pore-water in the Laurentian Trough. *Canadian Journal of Fisheries and Aquatic Sciences* 50: 1794-800.

MACTEC Engineering and Consulting, Inc. 2007a. ESPP, Olin Chemicals, McIntosh Plant Site, McIntosh, Alabama. Prepared for Olin Corporation, Charleston, Tennessee. June 8, 2007.

MACTEC Engineering and Consulting, Inc. 2007b. Work Plan for Mercury Methylation Research, Operable Unit 2, McIntosh Alabama. May 11, 2007.

MACTEC Engineering and Consulting, Inc. 2008a. Treatability Study Work Plan Cap Material Assessment, Operable Unit 2, McIntosh Alabama (April 1, 2008).

MACTEC Engineering and Consulting, Inc. 2008b. Storm Event Sampling Plan, Addendum to the ESPP, Operable Unit 2, McIntosh, Alabama (February 5, 2008).

MACTEC Engineering and Consulting, Inc. 2008c. Enhanced Sedimentation Pilot Project, Mercury Methylation Research, Operable Unit 2, McIntosh, Alabama (September 18, 2008).

Standard Methods for Examination of Water and Wastewaters, 18th, 19th, and 21st Eds. (American Public Health Association [APHA], et al., 1992, 1995, 2005) and subsequent editions.

SW846, 1986. "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," Third Edition, November 1986 and its updates.

URS Corporation. 2007. Semiannual Effectiveness Report, Second Semiannual Period for 2007, Olin Chemicals, McIntosh Plant Site, McIntosh, Alabama. Prepared for Olin Corporation, Charleston, Tennessee (November 2007).

U.S. Environmental Protection Agency, 1983. "Methods for Chemical Analysis of Water and Wastes," EPA-600/4-79-020, March 1983 and subsequent revisions.

U.S. Environmental Protection Agency, 1990. McIntosh Superfund Consent Order, Docket No. 90-13-C, Administrative Order by Consent for Remedial Investigation Feasibility Study, May 9, 1990.

U.S. Environmental Protection Agency, 1994. EPA Requirements for Quality Assurance Project Plans for Environmental Data Operation, EPA QA/R-5.

U.S. Environmental Protection Agency, 1998. EPA Guidance for Quality Assurance Project Plans, EPA QA/G-5.

U.S. Environmental Protection Agency, 2000a. Guidance for the Data Quality Objectives Process, EPA QA/G-4, EPA/600/R-96/055. August, 2000.

U.S. Environmental Protection Agency, 2000b. Guidance for Data Quality Objective Process for Hazardous Waste Sites, EPA QA/G-4HW, EPA/600/R-00/007. January, 2000.

U.S. Environmental Protection Agency, 2000c. Guidance for Data Quality Assessments, Practical Methods for Data Analysis, EPA QA/G-9, EPA/600/R-96/084. July, 2000.

U.S. Environmental Protection Agency. 2005. Correspondence to Olin Corporation. Subject OU-2 Remedial Pilot Project – Enhanced Sedimentation, October 25, 2005.

U.S. Environmental Protection Agency. 2006. Guidance on Systematic Planning Using the Data Quality Objectives Process. Office of Environmental Information, Washington, D.C. EPA QA/G-4. EPA/240/B-06/001, February 2006.

United States Army Corps of Engineers. 1987. EM 1110-2-5027, Chapter 3, Laboratory Testing, Subsection 3-2, *Sediment Characterization Tests* and Subsection 3-3, *Settling Tests*, pages 3-1 through 3-18, September 1987.

Woodward-Clyde Consultants. 1990. *Remedial Investigation (RI)/Feasibility Study Work Plan.* McIntosh Plant Site, Olin Corporation, McIntosh, Alabama. December 1990.

Woodward-Clyde Consultants. 1992. *Remedial Investigation (RI)/Feasibility Study Phase III Sampling and Analysis Plan.* McIntosh Plant Site, Olin Corporation, McIntosh, Alabama. June 1992.

Woodward-Clyde Consultants. 1993. Remedial Investigation Report, Olin Chemicals, McIntosh Plant Site, McIntosh, Alabama. Prepared for Olin Corporation, Charleston, Tennessee. February 1993.

Woodward-Clyde Consultants, 1994a. Additional Ecological Studies of OU-2 Work Plan. McIntosh Plant Site, Olin Corporation, McIntosh, Alabama. May 1994.

Woodward-Clyde Consultants, 1994b. Additional Ecological Studies of OU-2. Volumes 1 and 2. McIntosh Plant Site, Olin Corporation, McIntosh, Alabama. November 1994.

Woodward-Clyde Consultants, 1995. Ecological Risk Assessment of Operable Unit 2. McIntosh Plant Site, Olin Corporation, McIntosh, Alabama. May 1995.

Woodward-Clyde Consultants. 1996. *Feasibility Study Operable Unit 2.* McIntosh Plant Site, Olin Corporation, McIntosh, Alabama. February 1996.

TABLES

TABLE 1-1

Sample Media and Parameters

Sample Media	Field Parameters	Laboratory Parameters	Responsible Laboratory
Ground Water	pH Conductivity Temperature Dissolved Oxygen ORP Turbidity	Total & Dissolved Mercury Total & Dissolved Methylmercury	Battelle
		Hexachlorobenzene DDT, DDE, &DDD	Pace
Surface Water	pH Conductivity Temperature Dissolved Oxygen ORP Turbidity	Total & Dissolved Mercury Total & Dissolved Methylmercury	Battelle
		Hexachlorobenzene DDT, DDE, &DDD Alkalinity DOC Sulfide Sulfate TDS TSS Hardness	Pace/AES (TSS only)
Sediment/Soil	pH ORP Temperature	Methylmercury AVS/SEM % Moisture Pore-Water Mercury Pore-Water Methylmercury	Battelle
		Total Mercury Hexachlorobenzene DDT, DDE, &DDD Iron Manganese Molybdenum Selenium % Moisture pH ORP Sulfide Sulfate TOC Grain Size Density	Pace
Asiatic Clams		Total Mercury % Moisture	Pace
		Methylmercury % Moisture	Battelle
Fish (Whole & Fillet)		Total Mercury Hexachlorobenzene DDT, DDE, &DDD % Lipids	Pace

Notes:
AVS/SEM = Acid Volatile Sulfides/Simultaneously Extracted Metals
DDD = 2,4' and 4,4'-dichlorodiphenyldichloroethane
DDE = 2,4' and 4,4'-dichlorodiphenyldichloroethylene
DDT = 2,4' and 4,4'-dichlorodiphenyltrichloroethane
DOC = Dissolved Organic Carbon
ORP = Oxidation/Reduction Potential (Redox)
TDS = Total Dissolved Solids
TOC = Total Organic Carbon
TSS = Total Suspended Solids
% = percent

PREPARED/DATE: JAH 5/1/08
CHECKED/DATE: WPB 5/14/08

TABLE 1-2

Method Selection Rationale

Media	Parameter	Method	Rationale for selection
Water	Total & Dissolved Mercury	EPA 1631E and 7470A	Determines presence or absence of the constituent of concern; method RL meets or is less than ADEM Ambient Water Quality Criteria
	Total & Dissolved Methylmercury	EPA 1630 Draft	Determines presence or absence of the constituent of concern; method RL meets or is less than ADEM Ambient Water Quality Criteria
	Hexachlorobenzene	EPA 8081A or SW 8270C	Determines presence or absence of the constituent of concern
	Iron/Manganese	EPA 3010A / 6010B	Depending on iron speciation, iron can stimulate iron-reducing bacteria to methylate mercury, and these bacteria can do so at a rate equivalent to sulfate-reducing bacteria (Fleming and Nelson, 2006). In addition, transport of mercury in aquatic sediments is aided by iron and manganese colloids, and these metals have been found to co-occur in mercury-contaminated sediments (Gobeil and Cossa 1993; Mason *et al.*, 1993)
	Molybdenum [(1)]	EPA 3010A / 6010B	Molybdate (as sodium molybdate [Na2MoO4]) has been documented to block mercury methylation (Compeau and Barth, 1985; Fleming et al, 2006; Gilmour et al, 1992) in sediments.
	Selenium	EPA 3010A / 6010B	High selenium levels inhibit bioaccumulation rates of methylmercury (Barkay, et al., 1997)
	DDT, DDE, &DDD	EPA 8081A	Determines presence or absence of the constituent of concern
	Alkalinity	EPA 310.1	Total alkalinity measures the capacity of a water sample to neutralize an acid (*i.e.* , its buffering capacity)
	DOC/TOC	SM 5310B	DOC and TOC serves as a food source for methylating bacteria, especially at the surface water/sediment interface
	Sulfide	EPA 9030	A product of sulfate reduction
	Sulfate	EPA 9038	Sulfate stimulates sulfate-reducing bacteria to methylate mercury. However, in high sulfate environments, methylmercury production by sulfate-reducing bacteria may be inhibited due to the build-up of sulfide, a product of sulfate reduction (Benoit, 1999)
	TDS/TSS	SM 2540C/SM 2540D	Measurement of solids load in surface water
	Hardness	SM 2340C	Total hardness measures the amount of metal ions, particularly calcium and magnesium, which occur in a water sample
Sediment/Soil	Total Mercury	EPA 7471A	Determines presence or absence of the constituent of concern
	Methylmercury	EPA 1630 Draft	Determines presence or absence of the constituent of concern
	AVS/SEM	Allan,et al., 1991	Where the concentration of acid-volatile sulfide (AVS) (sulfide released by dilute acid treatment of moist sediment) exceeds the sum of the simultaneously extracted metals (SEM) from the same treatment, the excess sulfide will bind metals in insoluble complexes and hence biologically unavailable forms (Environment Australia, 2002)
	Pore-Water Mercury	SM 8080(M)/EPA 1631E	Determines the presence or absence of the constituent of concern in the aqueous phase within sediment
	Pore-Water Methylmercury	SM 8080(M)/EPA 1630 Draft	Determines the presence or absence of the constituent of concern in the aqueous phase within sediment
	Hexachlorobenzene	EPA 8081A or SW 8270C	Determines presence or absence of the constituent of concern
	DDT, DDE, &DDD	EPA 8081A	Determines presence or absence of the constituent of concern
	Iron/Manganese	EPA 3010A / 6010B	Depending on iron speciation, iron can stimulate iron-reducing bacteria to methylate mercury, and these bacteria can do so at a rate equivalent to sulfate-reducing bacteria (Fleming and Nelson, 2006). In addition, transport of mercury in aquatic sediments is aided by iron and manganese colloids, and these metals have been found to co-occur in mercury-contaminated sediments (Gobeil and Cossa 1993; Mason *et al.*, 1993)
	Molybdenum [(1)]	EPA 3050B / 6010B	Molybdate (as sodium molybdate [Na2MoO4]) has been documented to block mercury methylation (Compeau and Barth, 1985; Fleming et al, 2006; Gilmour et al, 1992) in sediments.
	Selenium	EPA 3050B / 6010B	High selenium levels inhibit bioaccumulation rates of methylmercury (Barkay, et al., 1997)
	% Moisture	ASTM D2216, D 2974-87, Freeze Dry	Needed to report results in sediments and/or soil to dry weight values
	pH	EPA 9045	General water quality parameter
	ORP	ASTM D1498-76	ORP is used to determine if a reducing or oxidizing condition is present
	TOC	EPA 9060	TOC serves as a food source for sulfate-reducing bacteria, which methylate mercury as a byproduct of sulfate reduction.
	Grain Size	ASTM D422	Grain size determines composition of particles in the sediments /soil.
	Density	ASTM D854	Physical parameter used to evaluate cap materials
Clams/Fish	Total Mercury	EPA 245.6	Determines presence or absence of the constituent of concern
	Methylmercury	EPA 1630 Draft	Determines presence or absence of the constituent of concern
	Hexachlorobenzene	EPA 8081A	Determines presence or absence of the constituent of concern
	DDT, DDE, &DDD	EPA 8081A	Determines presence or absence of the constituent of concern
	% Lipids	Bligh-Dyer, 1959	Needed to report results in tissue to dry weight values
	% Moisture	ASTM D2216, D 2974-87, Freeze Dry	Needed to report results in tissue to dry weight values

Notes:

[(1)] The total molybdenum reported will be used to calculate maximum molybdate concentration
AVS/SEM = Acid Volatile Sulfides/Simultaneously Extracted Metals
DDD = 2,4' and 4,4'-dichlorodiphenyldichloroethane
DDE = 2,4' and 4,4'-dichlorodiphenyldichloroethylene
DDT = 2,4' and 4,4'-dichlorodiphenyltrichloroethane
DOC = Dissolved Organic Carbon
ORP = Oxidation/Reduction Potential (Redox)
TDS = Total Dissolved Solids
TOC = Total Organic Carbon
TSS = Total Suspended Solids
% = percent

PREPARED/DATE: DWK 5/16/08
CHECKED/DATE: JAH 5/19/08

TABLE 1-3

Laboratory Precision and Accuracy Criteria

ANALYTE	LABORATORY	METHOD #	UNITS	MATRIX	MDL*	RL*	ACCURACY MS/MSD (%REC)	PRECISION (%RPD)	ACCURACY LCS (OPR) (%REC)	ADEM AMBIENT WATER QUALITY CRITERIA[1]
Total Mercury	Pace	EPA 245.2[12] or 7470A[3]	µg/L	Water	0.011	0.20	75-125	20	80-120	--
	Battelle	EPA 1631 E[11]	ng/L	Water	0.10	11	65-135	24	77-123	12 [(a)]
	Pace	EPA 245.5[10] or 7471A[3]	mg/kg (dw)	Sediment/Soil	0.002	0.020	75-125	20	80-120	--
	Pace	EPA 245.6[7]	mg/kg (ww)	Clams/Fish	0.00467	0.0167	70-130	10	85-115	--
Methylmercury	Battelle	Bloom, 1989 (EPA 1630 draft)[5,8]	ng/L	Water	0.02	0.05	65-135	35	67-133	4 [(b)]
	Battelle	Bloom, 1989 (EPA 1630 draft)[5,8]	ng/g (dw)	Sediment/Soil	0.03	0.10	65-135	35	67-133	--
	Battelle	Bloom, 1989 (EPA 1630 draft)[5,8]	µg/g (ww)	Clams	0.001	0.002	65-135	35	67-133	--
Pore-Water Mercury	Battelle	SM 8080[14]/EPA 1631E[11]	ng/L	Pore water	0.10	0.50	65-135	24	77-123	--
Pore-Water Methylmercury	Battelle	SM 8080[14]/EPA 1630 Draft[5,8]	ng/L	Pore water	0.020	0.050	65-135	35	67-133	--
Iron	Pace	EPA 3050B/6010B-ICP-AES[3]	µg/g	Sediment/Soil	2.6	10	10 - 235	20	65 - 134	--
	Pace	EPA 3010A/6010B-ICP-AES[3]	mg/L	Water	0.051	0.1	36 - 159	20	80 -124	1.0 [(a), (c)]
Manganese	Pace	EPA 3050B/6010B-ICP-AES[3]	µg/g	Sediment/Soil	0.059	1.5	10 - 200	20	72 - 122	--
	Pace	EPA 3010A/6010B-ICP-AES[3]	mg/L	Water	0.0013	0.015	47 - 147	20	86 - 119	--
Molybdenum	Pace	EPA 3050B/6010B-ICP-AES[3]	µg/g	Sediment/Soil	0.098	5	36 - 141	20	71 - 130	--
	Pace	EPA 3010A/6010B-ICP-AES[3]	mg/L	Water	0.0021	0.05	67 -140	20	54 - 142	--
Selenium	Pace	EPA 3050B/6010B-ICP-AES[3]	µg/g	Sediment/Soil	1.859	3.5	65-121	20	46-129	--
	Pace	EPA 3010A/6010B-ICP-AES[3]	mg/L	Water	0.01714	0.035	86-119	20	62-134	--
Hexachlorobenzene	Pace	EPA 3520C/8270C[3]	µg/L	Water	**1.07**	**10**	40 - 111	20	52 - 115	0.0003 [(b)]
	Pace	EPA 3541/8081A[3]	µg/L	Water	0.0003	**0.001**	NE	NA	NE	0.0003 [(b)]
	Pace	EPA 3550B/8270C[3]	mg/kg	Sediment/Soil	0.052	0.33	11 - 120	20	42 - 111	--
	Pace	EPA 3550B/8081A[3]	mg/kg	Sediment/Soil	0.00076	0.0016	NE	NA	NE	--
	Pace	EPA 3540C/8081A[3]	mg/kg	Clams/Fish	2.1	2.5	70-130	40	70-130	--
4,4'-DDD	Pace	EPA 3520C/8081A[3]	µg/L	Water	**0.023**	**0.1**	50-150	40	34-164	0.001 [(a)(c)]
4,4'-DDE	Pace	EPA 3520C/8081A[3]	µg/L	Water	**0.023**	**0.1**	50-150	40	47-142	--
4,4'-DDT	Pace	EPA 3520C/8081A[3]	µg/L	Water	**0.026**	**0.1**	50-150	40	41-154	--
4,4'-DDD	Pace	EPA 3550B/8081A[3]	mg/kg	Sediment/Soil	0.00106	0.0033	37-140	32	60-120	--
4,4'-DDE	Pace	EPA 3550B/8081A[3]	mg/kg	Sediment/Soil	0.00106	0.0033	49-128	37	70-127	--
4,4'-DDT	Pace	EPA 3550B/8081A[3]	mg/kg	Sediment/Soil	0.00107	0.0033	31-129	45	60-122	--
4,4'-DDD**	Pace	EPA 3540C/8081A[3]	mg/kg	Clams/Fish	0.99	5	60-133	33	62-132	--
4,4'-DDE**	Pace	EPA 3540C/8081A[3]	mg/kg	Clams/Fish	1.3	5	41-162	27	55-152	--
4,4'-DDT**	Pace	EPA 3540C/8081A[3]	mg/kg	Clams/Fish	0.77	5	61-129	40	55-132	--
2,4'-DDD	Pace	EPA3520C/8081A[3]	µg/L	Water	0.0083	0.05	--	--	--	--
2,4'-DDE	Pace	EPA3520C/8081A[3]	µg/L	Water	0.018	0.05	--	--	--	--
2,4'-DDT	Pace	EPA3520C/8081A[3]	µg/L	Water	0.0098	0.05	--	--	--	--
2,4'-DDD	Pace	EPA3550C/8081A[3]	mg/kg	Sediment/Soil	0.0014	0.00167	70-130	40	70-130	--
2,4'-DDE	Pace	EPA3550C/8081A[3]	mg/kg	Sediment/Soil	0.00077	0.00167	70-130	40	70-130	--
2,4'-DDT	Pace	EPA3550C/8081A[3]	mg/kg	Sediment/Soil	0.000052	0.00167	70-130	40	70-130	--
Alkalinity	Pace	EPA 310.1[2]	mg/L	Water	NA	5	--	20	--	--
Sulfate	Pace	EPA 9038[3]	mg/L	Water	0.5	0.5	75-125	20	85-115	--
Sulfide	Pace	EPA 9030[3]	mg/L	Water	**0.5**	**0.5**	75-125	20	85-115	0.002 [(a)(c)]
TDS	Pace	SM 2540 C[15]	mg/L	Water	NA	4	NA	20	80 - 120	--
TSS	Pace	SM 2540 D[15]	mg/L	Water	NA	4	NA	20	80 - 120	--
TOC/DOC	Pace	SM 5310 B[15]	mg/L	Water	0.45	1	75 - 125	20	90 - 110	--
TOC	Pace	SM 5310B[15]/ EPA 9060A[3]	%	Sediment/Soil	0.01	0.05	75-125	20	85-115	--
Density	Pace	SM 2710F Mo[15]	g/cm3	Sediment/Soil	--	0.5	--	20	97-103	--
Grain Size	Pace	ASTM D422 M/PSEP[9]	%	Sediment/Soil	0.01	0.01	--	20	--	--

TABLE 1-3

Laboratory Precision and Accuracy Criteria

ANALYTE	LABORATORY	METHOD #	UNITS	MATRIX	MDL*	RL*	ACCURACY MS/MSD (%REC)	PRECISION (%RPD)	ACCURACY LCS (OPR) (%REC)	ADEM AMBIENT WATER QUALITY CRITERIA[1]
pH	Pace	EPA 150.1[2]	pH units	Water	--	0.1	--	20	+/- 0.15	--
	Pace	EPA 9045 [3]	pH units	Sediment/Soil	--	0.1	--	20	+/- 0.15	--
AVS/SEM	Battelle	Allan,et al., 1991[13]	µmole/g	Sediment	0.01	0.05	75-125	25	75-125	--
Hardness	Pace	SM 2340 C[15]	mg/L	Surface Water	NA	10	--	20	90 - 110	--
% Moisture- PACE	Pace	ASTM D 2974-87[9]	%	Soil/Sediment	0.1	0.1	--	20	--	--
% Moisture - Battelle	Battelle	Freeze Dry or ASTM D2216[9]	%	Soil/Sediment	0.1	0.1	--	20	--	--
% Moisture - PACE	Pace	SM 2540G[15]	%	Clams/Fish	--	--	--	5	--	--
% Lipids	Pace	Bligh-Dyer, 1959[4]	%	Clams/Fish	0.1	0.5	--	25	--	--

Notes:
ADEM = Alabama Department of Environmental Management
AVS/SEM = acid volatile sulfides/simultaneously extracted metals
DDD = dichlorodiphenyldichloroethane
DDE = dichlorodiphenyldichloroethylene
DDT = dichlorodiphenyltrichloroethane
DOC = dissolved organic carbon
LCS = laboratory control sample
NA = not applicable
NE = not established
MDL = method detection limit
MS/MSD = matrix spike/matrix spike duplicate
REC = recovery
RL = reporting limit
OPR = ongoing precision and recovery (comparable to the LCS and is used for 1600 series)
ORP = Oxidation/Reduction Potential (Redox)
TDS = Total Dissolved Solids
TOC = Total Organic Carbon
TSS = Total Suspended Solids
% = percent
* MDL and RL values are based on routine method requirements for sample analysis and is reported on a wet weight basis with no dilutions.
** MDLs and RLs presented are for the 4,4' Isomer. The MDLs and RLs for the 2,4' Isomer have not been developed.

[1]Source: ADEM Water Quality Standards; ADEM Water Division - Water Quality Program, May 2007

[2]EPA. 1979. Revised (1983). Methods for the Chemical Analysis of Water and Wastes. EPA-600/4-79-020. Environmental Monitoring Systems Laboratory, Cincinnati, OH.

[3]EPA. 1996a. Test Methods for Evaluating Solid Waste. EPA, Office of Solid Waste and Emergency Response, Washington, D.C. (SW846)

[4]Bligh, E.G., and W. J. Dyer. 1959. A Rapid Method of Total Lipid Extraction and Purification. Canadian Journal of Biochemistry and Physiology. Vol 37 No. 8. pp. 911-917.

[5]Bloom 1989. Determination of Picogram Levels of Methylmercury by Aqueous Phase Ethylation, Followed by Cryogenic Gas Chromatography with Cold Vapor Atomic Fluorescence Detection*Can. J. Fish.*

[6]Plumb, R.H., Jr. 1989. Procedure for Handling and Chemical Analysis of Sediment and Water Samples. Tech. Rep. EPA/CE-81-1. Prepared by Great Lakes Laboratory, State University College at Buffalo, Buffalo, NY, for the EPA/U.S. Army Corps of Engineers Technical Committee on Criteria for Dredged and Fill Material. U.S. Army Engineer Waterways Experiment Station, Vicksburg, MS.

[7]EPA. 1991. Method 245.6. Mercury in Tissues by Cold Vapor (CV/AAS). April 1991.

[8]EPA. 1998a. Method 1630. Methyl Mercury in Water by Distillation, Aqueous Ethylation, Purge and Trap, and CVAFS. Draft. March 1998.

[9]ASTM. 1987, 1998, 2007. Annual Book of American Society for Testing and Materials (ASTM) Standards.

[10]EPA. 1999. Method 245.5. Standard Operating Procedure for the Analysis of Mercury in Sediment and Solids. December 1999.

[11]EPA. 2001. Method 1631E. Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry. EPA 821-R-01-024. August 2002.

[12]EPA. 2001. Method 245.2. Mercury in Water by Cold Vapor Atomic Adsorption Spectrometry. April 1991.

[13]Allen, H.E., F. Gongmin, W. Boothman, D. DiToro, and J. Mahoney. 1991. Determination of Acid Volatile Sulfides (AVS) and Simultaneously Extracted Metals in Sediment. Draft Analytical Method for Determination of Acid Volatile Sulfide in Sediment. USEPA Office of Science and Technology, Washington, DC. April, 1991.

[14] SM. 1997. Method 8080. Sediment Porewater Testing (filtration procedure). Standard Methods, 20th Edition.

[15] Standard Methods for Examination of Water and Wastewaters, 18th, 19th and 21st Eds. (American Public Health Association [APHA], et al., 1992, 1995, 2005) and subsequent editions

(a) Region 4 screening value (2001); Source: http://www.epa.gov/region4/waste/ots/ecolbul.htm#tbl1
(b) USEPA Region 3 Freshwater Screening Benchmark Source: http://www.epa.gov/reg3hwmd/risk/eco/btag/sbv/fw/screenbench.htm
(c) National Ambient Water Quality Criteria (2005), Source: http://www.epa.gov/waterscience/criteria/wqcriteria.html

= MDL and/or RL exceeds AWQC

PREPARED/DATE: JAH 5/1/08
CHECKED/DATE: WPB 10/9/08

TABLE 1-4

Required Containers, Preservation Techniques, Temperature Requirements, and Holding Times

Sample Matrix	Laboratory Parameters	Container[1]	Preservative	Temperature Requirment	Maximum Holding Time	Responsible Laboratory
GROUND WATER	Total Low-Level Mercury	1 liter T or G	Zero headspace/Upon lab arrival preserve with Bromine monochloride	Wet ice/cool 4°C	48 hours prior to preservation and 90 days after preservation	Battelle
	Dissolved Low-Level Mercury		Zero headspace/Upon lab arrival, filter w/0.45 µm capsule filter then preserve with Bromine monochloride		48 hours prior to preservation and 90 days after preservation	
	Methylmercury		Zero headspace/Upon lab arrival preserve with HCl to a pH< 2		48 hours prior to preservation and 6 months after preservation	
	Hexachlorobenzene	2 x 1 liter amber G with PTFE-lined lids	None	Wet ice/cool 4°C	7 days until extraction and analyze within 40 days after extraction	Pace
	DDT, DDE, & DDE	2 x 1 liter amber G with PTFE-lined lids	None	Wet ice/cool 4°C	7 days until extraction and analyze within 40 days after extraction	
SURFACE WATER	Total Low-Level Mercury	1 liter T or G	Zero headspace/Upon lab arrival preserve with Bromine monochloride	Wet ice/cool 4°C	48 hours prior to preservation and 90 days after preservation	Battelle
	Dissolved Low-Level Mercury		Zero headspace/Upon lab arrival, filter w/0.45 µm capsule filter then preserve with Bromine monochloride		48 hours prior to preservation and 90 days after preservation	
	Methylmercury		Zero headspace/Upon lab arrival preserve with HCl to a pH< 2		48 hours prior to preservation and 6 months after preservation	
	Hexachlorobenzene	2 x 1 liter amber G with PTFE-lined lids	None	Wet ice/cool 4°C	7 days until extraction and analyze within 40 days after extraction	Pace/AES[3]
	DDT, DDE, & DDE	2 x 1 liter amber G with PTFE-lined lids	None	Wet ice/cool 4°C	7 days until extraction and analyze within 40 days after extraction	
	Alkalinity	500 ml HDPE	None	Wet ice/cool 4°C	14 days	
	DOC		None; Filter in lab and preserve with H_2SO_4 to a pH < 2		28 days	
	TDS		None		7 days	
	TSS					
	Sulfate	250 ml HDPE or G	None	Wet ice/cool 4°C	28 days	
	Sulfide by EPA 376.1	250 ml HDPE or G	Zinc Acetate and Sodium Hydroxide to pH>12	Wet ice/cool 4°C	7 days	
	Hardness	250 ml HDPE or G	HNO_3 to pH<2	Wet ice/cool 4°C	6 months	

TABLE 1-4

Required Containers, Preservation Techniques, Temperature Requirements, and Holding Times

Sample Matrix	Laboratory Parameters	Container[1]	Preservative	Temperature Requirment	Maximum Holding Time	Responsible Laboratory
SEDIMENTS/SOIL	Methylmercury	1 x 8 oz G	None	Dry Ice[2]	28 days	Battelle
	AVS/SEM				14 days	
	Total Mercury	1 x 4 oz G	None	Wet ice/cool 4°C	28 days	Pace
	Iron, Manganese, Molybdenum, Selenium	1 x 4 oz G			6 months	
	Sulfate	1 x 4 oz G			28 days	
	Sulfide	1 x 4 oz G			7 days	
	Hexachlorobenzene & DDTr	1 x 4 oz G 1 x 4 oz G 1 x 8 oz G			14 days until extraction and analyze within 40 days after extraction	
	% Moisture				None Established	
	TOC	1 x 4 oz G	None	Wet ice/cool 4°C	28 days	
	Density	1x 8 oz G			None Established	
	Grain Size	1 x 32 oz G				
ASIATIC CLAMS	Methylmercury	10 g (20-30 clams) ZB	None	Dry Ice[2]	Frozen -6 months Freeze dried - unlimited	Battelle
	% Moisture				None Established	
	Total Mercury	10 g (20-30 clams) ZB	None	Dry Ice[2]	Frozen -6 months Freeze dried - unlimited	Pace
	% Moisture				None Established	
FISH (fillet)	Total Mercury	120 g ZB	None	Dry Ice[2]	Frozen -6 months Freeze dried - unlimited	Pace
	Hexachlorobenzene					
	DDT, DDE, & DDE					
	% Lipids					

Notes:

[1]T = Teflon®, ZB = ziplock bag, HPDE = high density polyethylene , G = glass

[2]Wet ice can be substituted if needed.

[3]AES is responsible for analyzes of TSS in ISCO samples only

PTFE = polytetrafluoroethylene (Teflon®)
AVS/SEM = Acid Volatile Sulfides/Simultaneously Extracted Metals
DDD = 2,4' and 4,4'-dichlorodiphenyldichloroethane
DDE = 2,4' and 4,4'-dichlorodiphenyldichloroethylene
DDT = 2,4' and 4,4'-dichlorodiphenyltrichloroethane
DDT r = Total DDD, DDE, and DDT residues
DOC = Dissolved Organic Carbon
TDS = Total Dissolved Solids
TOC = Total Organic Carbon
TSS = Total Suspended Solids
% = percent

PREPARED/DATE: JAH 5/1/08
CHECKED/DATE: WPB 5/24/08

TABLE 2-1

Proposed Number of Field and QC Samples per OU2 Work Plan Sampling Event

Enhanced Sedimentation Pilot Project				
Sample Medium	**Number***	**Analyses***	**Analytical Methods***	**QA/QC***
Surface Water	20	Mercury (unfiltered)	USEPA Method 1631E	2 duplicates
		Mercury (filtered)	USEPA Method 1631E	1 MS/MSD
		Methylmercury (unfiltered)	EPA 1630 (draft)by extraction	2 Field Blanks
		Methylmercury (filtered)	EPA 1630 (draft) by extraction	1 Rinsate Blank
		Hardness as $CaCO_3$	USEPA Method 130.2	
		Total Alkalinity	USEPA Method 310.1	
		Sulfide	USEPA Method 9030	
		Sulfate	USEPA Method 9038	
		DOC	USEPA Method 9060A	
		TDS	USEPA Method 160.1	
		TSS	USEPA Method 160.2	
Surficial Sediment	39	Mercury	USEPA Method 7471	4 duplicates
		Methylmercury	EPA 1630 (draft) by extraction	2 MS/MSD
		DDTr[b]	USEPA Method 8081A	1 Rinsate Blank
		Hexachlorobenzene[b]	USEPA Method 8081A	
		AVS/SEM	Allen, et al., 1991/EPA 1638	
		Fe/Mn/Mo[b]/Se[b]	USEPA Method 6010B	
		Percent moisture	Freeze Drying	
		Sulfide	USEPA Method 9030	
		Sulfate	USEPA Method 9038	
		TOC	EPA 9060A	
		Grain size	ASTM D422 M/PSEP	
		Percent moisture	ASTM D2216	
		Density	ASTM D854	
Clam Tissue	10	Mercury	USEPA Method 245.6	1 duplicate
		Methylmercury	EPA 1630 (draft) by extraction	1 MS/MSD
		Percent lipid	Bligh-Dyer, 1959	
		Percent moisture	Freeze Dry	
Sediment Trap	48	Mercury	USEPA Method 7471	
		TOC[b]	EPA 9060A	
		Grain size	ASTM D422 M/PSEP	
		Density	ASTM D854	
		Percent moisture[b]	ASTM D2216	
		TSS[b]	USEPA Method 160.2	
		Volume in trap[b]		
Soil bed	4	Mercury	USEPA Method 7471	

Treatability Study				
Sample Medium	**Number***	**Analyses***	**Analytical Methods***	**QA/QC***
Surface Water	60	Mercury (unfiltered)	USEPA Method 1631	6 Duplicates
		Methylmercury (unfiltered)	EPA 1630 (draft)	3 MS/MSD
				3 Field Blanks
				1 Rinsate Blank
	120	TSS	USEPA Method 160.2	12 Duplicates
Sediment	50	Mercury	USEPA Method 7471	5 Duplicates
		Methylmercury	EPA 1630 (draft) by extraction	3 MS/MSD
		TOC	USEPA 9060A	1 Rinsate Blank
		Grain Size	ASTM D422 M/PSEP	
		% Moisture	ASTM D2216	
		Density	ASTM D854	
Asiatic Clams	11	Mercury	USEPA Method 245.6	2 Duplicates
		Methylmercury	EPA 1630 (draft) by extraction	1 MS/MSD

Groundwater Investigation				
Sample Medium	**Number***	**Analyses***	**Analytical Methods***	**QA/QC***
Groundwater	14	Mercury (unfiltered)	USEPA Method 1631E	2 Duplicates
		Mercury (filtered)	USEPA Method 1631E	1 MS/MSD
				1 Field Blank
				1 Rinsate Blank

Storm Event Surface Water Sampling				
Sample Medium	**Number***	**Analyses***	**Analytical Methods***	**QA/QC***
Surface Water	18 (per storm event)	TSS	USEPA Method 160.2	2 Duplicates
		Grain size fraction	Long Tube Method [a]	
ISCO Surface Water	18 (per storm event)	TSS	USEPA Method 160.2	2 Duplicates

Notes:

[a] Long Tube Testing Report (MACTEC, September 22, 2006) method using vacuum filtration

[b] Analyses were added to the ESPP for the follow-on annual sampling events

ASTM - American Standard Test Methods
AVS/SEM - acid-volatile sulfide/simultaneously extracted metals
$CaCO_3$ - calcium carbonate
DOC - dissolved organic carbon
Fe - iron
Mn - manganese
Mo - molybdenum
MS/MSD - matrix spike/matrix spike duplicate
QA/QC - quality assurance/quality control
Se - selenium
TDS - total dissolved solids
TOC - total organic carbon
TSS - total suspended solids
USEPA - United States Environmental Protection Agency
*The number of samples and analyses presented on this table may differ from actual events.

PREPARED/DATE: DWK 05/15/08
CHECKED/DATE: JAH 5/19/08

TABLE 2-2

QC SAMPLES AND FREQUENCY OF ANALYSIS FOR INORGANICS

	Metals by ICP Methods 6010B, 200.7	**Metals by ICPMS Methods 6020A, 1638, 200.8**	**Mercury by Methods 7470A, 7471A, 245.1, 245.6**	**Low-Level Mercury by Method 1631E**	**Methylmercury by Method 1630 Draft**	**Other Inorganics**
LABORATORY QC SAMPLES						
Initial Calibration Verification (ICV)	Daily and prior to sample analysis.	Daily and prior to sample analysis.	Daily and prior to sample analysis.	Daily and prior to sample analysis.	Daily and prior to sample analysis.	After every calibration curve.
Continuing Calibration Verification (CCV)	One per every 10 samples analyzed.	One per every 10 samples analyzed.	One per every 10 samples analyzed.	One per every 10 samples analyzed.	One per every 10 samples analyzed.	One per every 10 samples analyzed.
Continuing Calibration Blank (CCB)	One per every 10 samples analyzed.	One per every 10 samples analyzed.	One per every 10 samples analyzed.	One per every 10 samples analyzed.	Not applicable.	One per every 10 samples analyzed.
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	For each matrix, one pair per batch or 20 samples, whichever is most frequent.	For each matrix, one pair per batch or 20 samples, whichever is most frequent.	For each matrix, one pair per batch or 20 samples, whichever is most frequent.	For each matrix, one pair per batch or 20 samples, whichever is most frequent.	For each matrix, one pair per batch or 20 samples, which ever is most frequent.	When applicable, for each matrix, one pair per batch or 20 samples, whichever is most frequent. Otherwise, one duplicate at that rate.
Method Blank	For each matrix, one per batch or 20 samples, which ever is most frequent.	For each matrix, one per batch or 20 samples, whichever is most frequent.	For each matrix, one per batch or 20 samples, whichever is most frequent.	Three per batch or 20 samples, whichever if most frequent.	For each matrix, three per 20 samples, which ever is most frequent	For each matrix, one per batch or 20 samples, whichever is most frequent.
ICP Interference Check Sample	At the beginning of each analytical sequence.	At the beginning of each analytical sequence.	Not applicable.	Not applicable.	Not applicable.	Not applicable.
Certified/Standard Reference Material (CRM/SRM)/Quality Control sample (QCS)	Not applicable	For methods 1638 & 200.8; QCS – Analyze Quarterly	For method 245.6 – one per batch	One per every 20 samples analyzed	One per every 20 samples analyzed	Not applicable.
Laboratory Control Sample (LCS)/Blank Spike (BS)/Ongoing	For each matrix, one per batch or 20 samples, whichever	For each matrix, one per batch or 20 samples, whichever	For each matrix, one per batch or 20 samples, whichever	OPR - One at beginning and end of batch	For each matrix, one per batch or 20 samples, whichever	For each matrix, one per batch or 20 samples, whichever

TABLE 2-2

QC SAMPLES AND FREQUENCY OF ANALYSIS FOR INORGANICS

	Metals by ICP Methods 6010B, 200.7	**Metals by ICPMS Methods 6020A, 1638, 200.8**	**Mercury by Methods 7470A, 7471A, 245.1, 245.6**	**Low-Level Mercury by Method 1631E**	**Methylmercury by Method 1630 Draft**	**Other Inorganics**
LABORATORY QC SAMPLES						
Precision and Recovery (OPR)	is most frequent.	is most frequent.	is most frequent.		is most frequent.	is most frequent.
Serial Dilution /Dilution test	For each matrix, one per batch.	For each matrix, one per batch.	For each matrix, one per batch.	Not applicable.	Not applicable.	Not applicable.
Post Digestion Spike (PDS)	For each matrix, one per batch.	For each matrix, one per batch.	Performed if serial dilution is out of control	Not applicable.	Not applicable.	Not applicable.
Internal Standards	Not Applicable	Every sample	Not Applicable	Not Applicable	Not Applicable	Not Applicable
Laboratory Replicate/Duplicate	For each matrix, one per batch or 20 samples, whichever is most frequent	For each matrix, one per batch or 20 samples, whichever is most frequent	For each matrix, one per batch or 20 samples, whichever is most frequent	One per batch or 20 samples, whichever is most frequent	For each matrix, one per batch or 20 samples, whichever is most frequent	For each matrix, one per batch or 20 samples, whichever is most frequent
FIELD QC SAMPLES						
Field Blank	None	None	None	Two per day for surface water and groundwater samples	Two per day for surface water and groundwater samples	None
Field Duplicate	For each matrix, one per 10 field samples or fraction thereof.	For each matrix, one per 10 field samples or fraction thereof.	For each matrix, one per 10 field samples or fraction thereof.	For each matrix, one per 10 field samples or fraction thereof.	For each matrix, one per 10 field samples or fraction thereof.	For each matrix, one per 10 field samples or fraction thereof.
Equipment Rinse Blank (non-dedicated)	One per sampling event per equipment type.	One per sampling event per equipment type.	One per sampling event per equipment type.	One per sampling event per equipment type.	One per sampling event per equipment type.	One per sampling event per equipment type.

PREPARED/DATE: JAH 05/09/08
CHECKED/DATE: WPB 5/25/08

TABLE 2-3

QC SAMPLES AND FREQUENCY OF ANALYSIS FOR ORGANICS

	Semi-volatile Organic Analysis	Pesticides
LABORATORY QC SAMPLES		
GC/MS Tuning	At the beginning of each 12-hour analytical shift.	Not applicable.
Initial Calibration	Initially and after continuing calibration verification fails to meet acceptance criteria.	Initially and after continuing calibration verification fails to meet acceptance criteria.
Continuing Calibration Verification (CCV)	At the beginning of each 12-hour analytical shift.	One after every 10 samples analyzed.
DDT/Endrin Degradation Check	Not applicable.	After each continuing calibration analysis.
Internal Standard(s)	Every sample.	Not applicable.
Surrogate(s)	Every sample.	Every sample.
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	For each matrix, one per batch or 20 samples, whichever is most frequent.	For each matrix, one per batch or 20 samples, whichever is most frequent.
Method Blank	For each matrix, one per batch or 20 samples, whichever is most frequent.	For each matrix, one per batch or 20 samples, whichever is most frequent.
Laboratory Control Sample (LCS)	For each matrix, one per batch or 20 samples, whichever is most frequent.	For each matrix, one per batch or 20 samples, whichever is most frequent.
FIELD QC SAMPLES		
Field Blank	Not applicable	Not applicable
Field Duplicate	For each matrix, one per 10 field samples or fraction thereof.	For each matrix, one per 10 field samples or fraction thereof.
Trip Blank	Not applicable.	Not applicable.
Equipment Rinse Blank (non-dedicated)	One per sampling event per equipment type.	One per sampling event per equipment type.

PREPARED/DATE: JAH 05/09/08
CHECKED/DATE: WPB 5/25/08

TABLE 2-4

ROUTINE PREVENTATIVE MAINTENANCE PROCEDURES AND SCHEDULES

Instrument	Maintenance Procedures/Schedule	Spare Parts in Stock
Gas Chromatograph/ Mass Spectrometer (GC/MS)	Replace pump oil as needed. Change septa weekly or as often as needed. Change gas line dryers as needed. Replace electron multiplier as often as needed Replace glass jet splitter as needed. Replace GC injector glass liner weekly or as often as needed. Replace GC column as needed. Check to ensure the gas supply is sufficient for the day's activity and the delivery pressures are set as described in the SOP. Check to ensure that the pressure on the primary regulator never drops below 100 psi.	Syringes Septa Various electronic parts Gas jet splitter GC column Glass liner
Gas Chromatograph (GC)	Change septa weekly or as often as needed. Change gas line dryers as needed. Replace GC injector glass liner weekly or as needed. Replace GC column as needed. Clean/replace GC detector as needed. Check to ensure the gas supply is sufficient for the day's activity, and the delivery pressures are set as described in the SOP. Check to ensure that the pressure on the primary regulator never drops below 100 psi.	Syringes Septa Detector supplies Glass liner GC column
Inductively Coupled Plasma	Change pump tubing daily or as often as needed. Check nebulizer daily. Clean optics biannually or as needed. Check to ensure the gas supply and liquid argon supply are sufficient for the day's activity, and the delivery pressures are set as described in the SOP. Check to ensure that the pressure on the primary regulator never drops below 100 psi. Clean torch every 3 months or as needed.	Pump tubing Nebulizer Argon
Cold Vapor Mercury Analyzer UV/Visible Spectrometer	Change absorbent as needed. Clean windows monthly. Maintenance, other than general cleaning and calibration is performed by service representative as needed.	

PREPARED/DATE: JAH 5/23/08
CHECKED/DATE: WPB 5/25/08

TABLE 2-5

FIELD CALIBRATION FREQUENCY AND CORRECTIVE ACTION PROCEDURES

SITUATION	CALIBRATION[(a)]	FREQUENCY	FIELD OBJECTIVE AFFECTED	CORRECTIVE ACTION PROCEDURE
Equipment malfunction pH Conductivity Temperature Dissolved Oxygen Redox Potential Turbidity	- Calibrate with two buffer solutions that bracket expected sample pH - Calibrate with two standards in expected range of sample SC - Calibrate within expected temperature range of samples - Calibrate per manufacturer's Instructions - Calibrate per manufacturer's Instructions - Check calibration within expected range of sample turbidity	- Prior, middlet end of day - Twice Daily - Prior to project by manufacturer - Twice Daily - Twice Daily - Twice Daily	Equipment is calibrated and operating properly	- Notification of site supervisory personnel - Repair or replace malfunctioning parts - Recalibrate and/or replace standards - Resample or repeat task if necessary - Document to Project Manager
Incorrect sample collection procedures	NA	NA	Samples are taken according to standard operating procedures.	- Notification of site supervisory personnel - Review of situation and correct procedures; recollect the sample - Document to Project Manager
Insufficient sample volume collection	NA	NA	Sufficient sample volume is provided to maintain sample integrity and so that all required analyses can be conducted.	- Notification of site supervisory personnel by laboratory manager - Review site affected and impact of samples on site characterization correct procedures; recollect sample if necessary - Document to Project Manager
Incorrect measurement data collection	NA	NA	Measurements are conducted according to standard operating procedures	- Notification of site supervisory personnel - Review of situation and correct procedures - Document to Project Manager and Quality Assurance Officer (QAO)

TABLE 2-5

FIELD CALIBRATION FREQUENCY AND CORRECTIVE ACTION PROCEDURES

SITUATION	CALIBRATION[(a)]	FREQUENCY	FIELD OBJECTIVE AFFECTED	CORRECTIVE ACTION PROCEDURE
Measurement Outside of Expected Range				
pH Conductivity Temperature Dissolved Oxygen Redox Potential	- 5 to 9 - .050 to 4 mS/cm - 15 to 31°C - 0 to 10 mg/L (Driscoll, 1986) - -800 to 400 mV	NA	Measurements are conducted according to standard operating procedures	- Notification of site supervisory personnel - Review of situation and correct procedures - Document to Project Manager and Quality Assurance Officer (QAO)

NA - Not Applicable

(a) For multi-parameter units, follow manufacturer's instructions.

PREPARED/DATE: JAH 05/15/08

CHECKED/DATE: WPB 5/24/08

TABLE 4-1

Data Qualification Flags

Flag	Positive Results	Non-Detect Results
FLAGS FOR DATA WITHIN ACCEPTABLE LIMITS (Usable as Reported)		
No Flag	Use datum without qualification	Use datum without qualification
FLAGS FOR DATA WITHIN ACTION LIMITS (Usable with Qualification)		
J	Estimated quantitation based upon QC data	Estimated quantitation based upon QC data
JB	Estimated quantitation: possibly biased high or false positive based upon blank data	(Not applicable)
JH	Estimated quantitation - possibly biased high based upon QC data	(Not applicable)
JL	Estimated quantitation - possibly biased low based upon QC data	Possible false non-detect based upon QC data
JQ	Estimated quantitation; value is between the reporting limit and the detection limit	(Not applicable)
UJ	(Not applicable)	Undetected; Reported detection limit is imprecise
UL	(Not applicable)	Undetected; Data biased low - Reported detection limit is higher than indicated
FLAGS FOR DATA OUTSIDE ACTION LIMITS (Unsable)		
R	Datum rejected based upon QC data: do not use	Datum rejected based upon QC data: do not use

Note that if the QC results suggest contradictory flags, the following hierarchy should be used to select the appropriate flag: to assign:

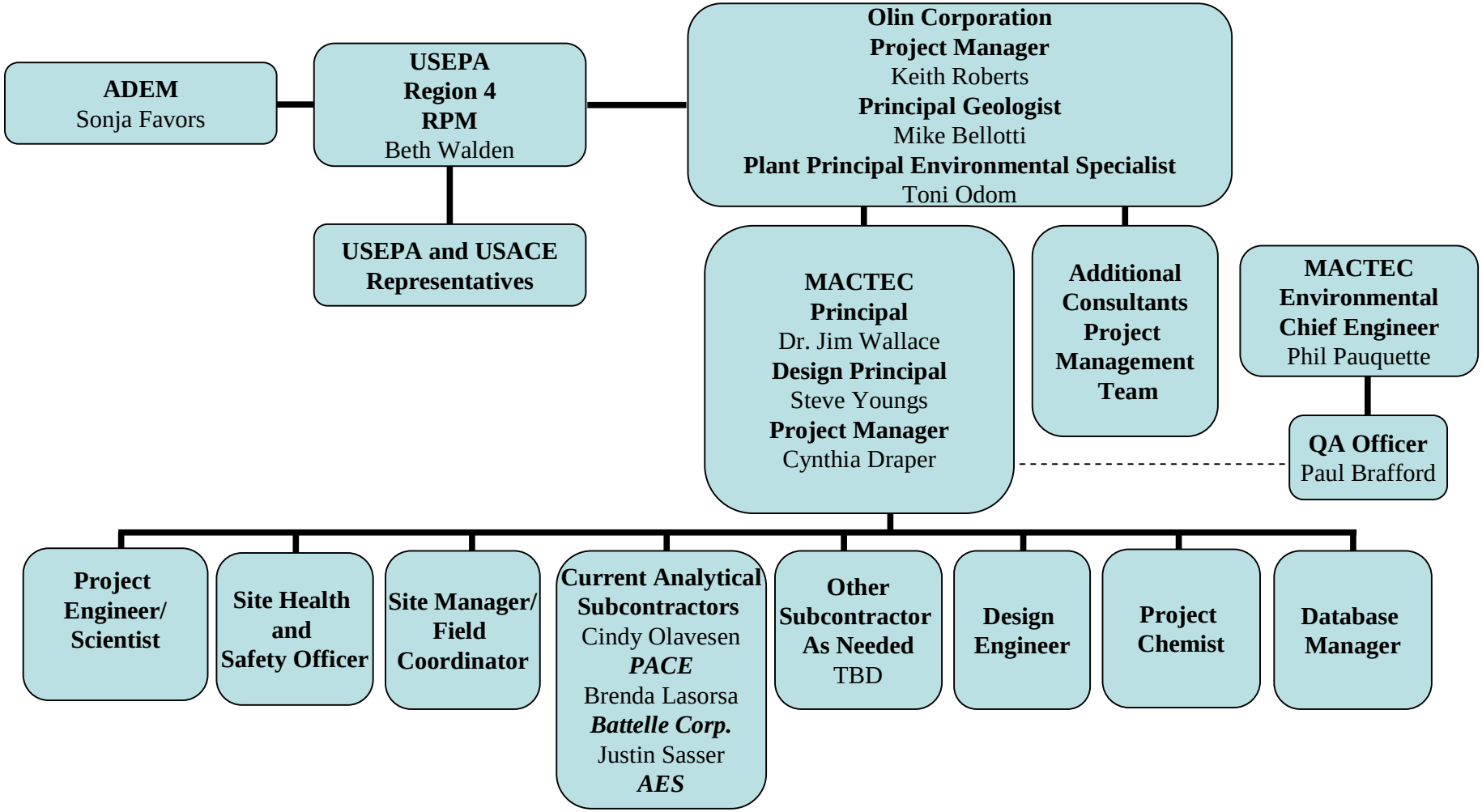
R>JB>JH>JL> JQ
JH + JL = J
JQ > J

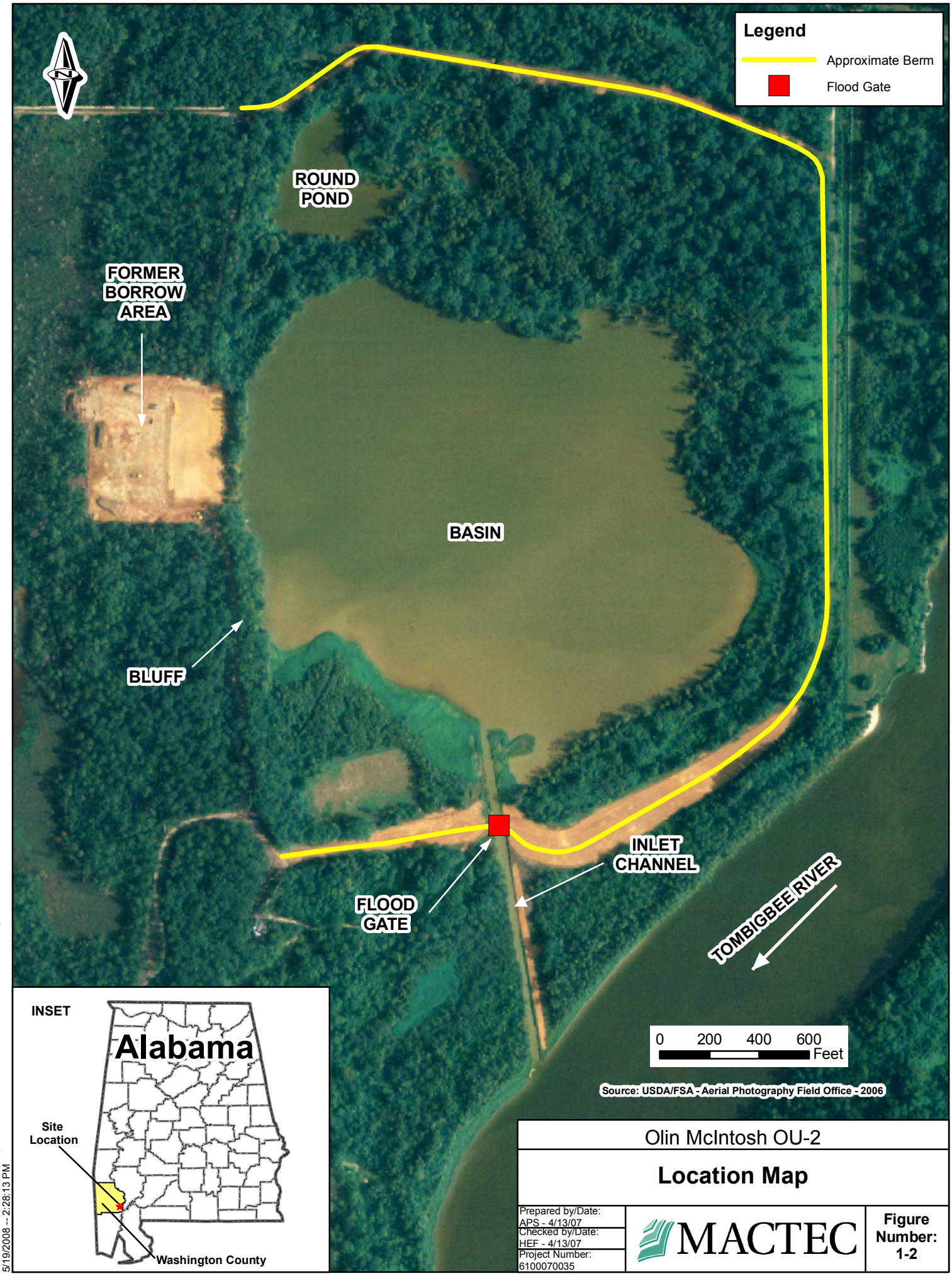
PREPARED/DATE: JAH 5/1/08
CHECKED/DATE: WPB 5/25/08

FIGURES

Figure 1-1

Olin OU 2 McIntosh
Project Organization



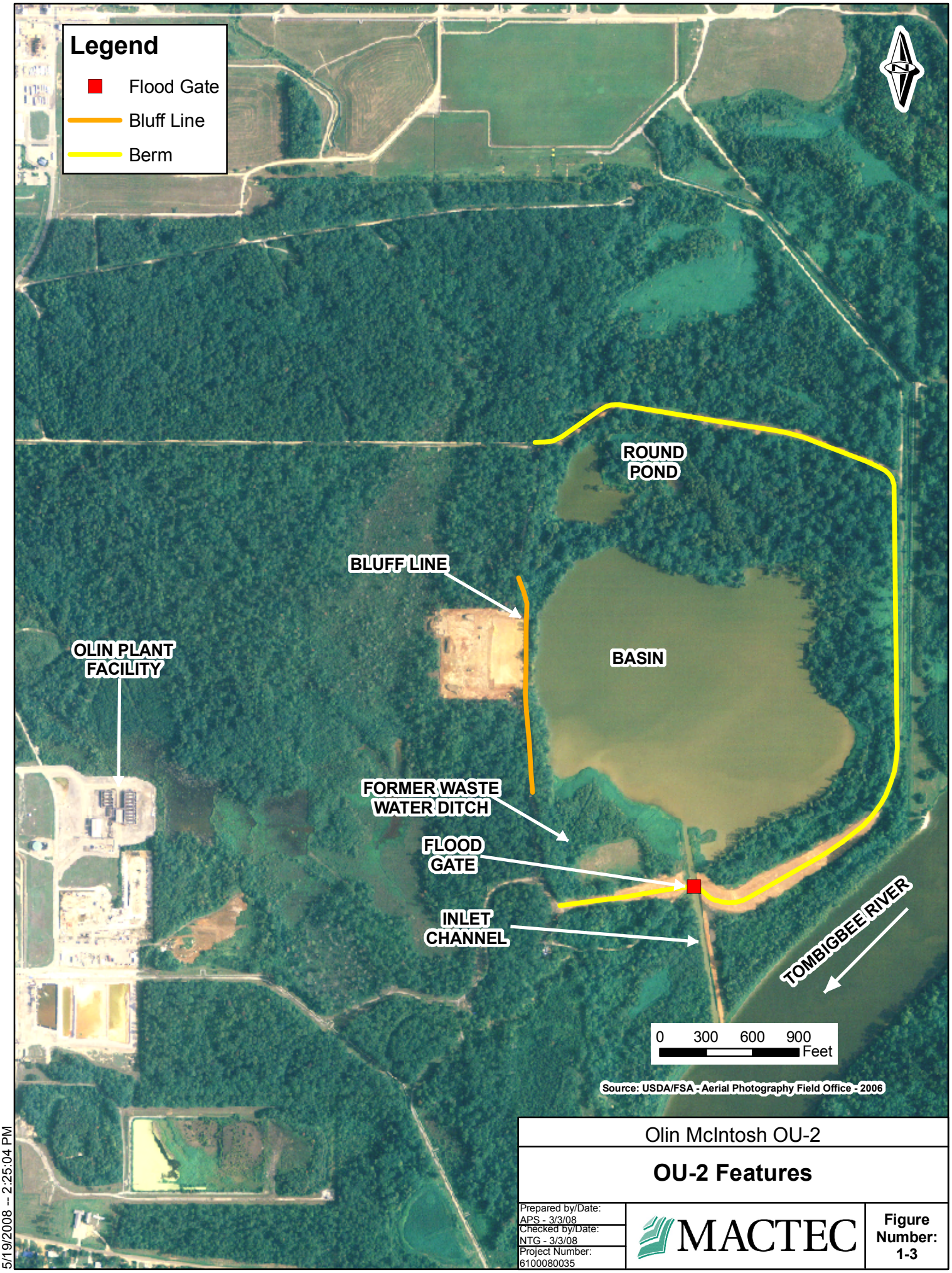


Olin McIntosh OU-2

Location Map

Prepared by/Date: APS - 4/13/07 Checked by/Date: HEF - 4/13/07 Project Number: 6100070035	MACTEC	Figure Number: 1-2

Map Document: (P:\gis\Projects2007\olin_mcintosh\mxds_3_21_07\site_loc_fig_1-2.mxd)
5/19/2008 -- 2:28:13 PM



Olin McIntosh OU-2

OU-2 Features

Prepared by/Date: APS - 3/3/08 Checked by/Date: NTG - 3/3/08 Project Number: 6100080035	MACTEC	Figure Number: 1-3

Map Document: (P:\gis\Projects2007\olin_mcintosh\csm_GROUNDWATER\Figure1-3 OU2 Features.mxd)
5/19/2008 -- 2:25:04 PM

Figure 3-1

No.:	**Nonconformance and Corrective Action Report (QAF 16-1)**
Organization:	Location:
Reported By:	Date:

Nonconformance

Description of Nonformance:
Representative Notified:
Date Notified: / Date Corrective Action Plan Due:

Corrective Action Plan

Description of Evaluation to Determine Assignable Cause:
Assignable Cause:
Potential Harm:
Description of Corrective Actions (current and to prevent recurrence):
Estimated Completion Date:
Recommended disposition of nonconforming items (i.e. reject/dispose, repair, rework, use-as-is) Include technical justification:

Signature:	**Date:**
Corrective Action Approval Signature:	**Date:**

Corrective Action Closure

Comments:
Approved/Actual Disposition of Nonconforming Items:

Approval Signature:	**Date:**

APPENDIX A

LOW LEVEL SAMPLING PROCEDURES

APPENDIX A

LOW-LEVEL METALS SAMPLING PROCEDURES

Surface water and groundwater samples to be analyzed for low-level mercury by U. S. Environmental Protection Agency (USEPA) Method 1631E and methylmercury by USEPA Method 1630 Draft will be collected using the "clean hands/dirty hands" sampling procedures specified in USEPA Method 1669.

A two-person team is required for sample collection. One member is designated as "Dirty Hands" and the other member is designated as the "Clean Hands". The "Dirty Hands" member is responsible for the preparation of the sampler (except the sample container itself), operation of any machinery, and all other activities that do not involve contact with the sample. The "Clean Hands" member will handle operations which involve contact with the sample bottle and transfer of the sample from the collection device to the sample bottle.

Each member will wear clean, lint-free outer clothing (such as Tyvek) and at least two pairs of non-talc gloves (wearing multiple layers of clean gloves will minimize disruption of sampling activities when gloves are to be changed out). "Clean Hands" should wear an additional shoulder length polyethylene pair of gloves. "Clean" sampling equipment and sample containers will be obtained from the laboratory responsible for the testing. For the Olin McIntosh OU 2 low-level mercury and methylmercury sampling, preservation and filtering will be performed at the laboratory.

Sample equipment used for low-level mercury sampling will be non-metallic or (when using pumps with some metal parts) the sample will not be allowed to come in direct contact with metal parts in the equipment. Sample containers for mercury will be made of fluoropolymer (FEP, PTFE, Teflon®) or glass because mercury vapors can diffuse in or out of other materials resulting in either contamination or biased-low results (Bloom, 1993).

Sample tubing will be composed of fluoropolymer or styrene/ethylene/butylene/silicone (SEBS) material. When sampling from a boat, the boat and oars should be made of wood or fiberglass and cleaned with water from the sampling site. Gasoline- or diesel-fueled motors should be avoided. If motors are

required then the engine should be shut off at a distance far enough from the sampling point to avoid contamination.

If mercury concentrations are known, samples are to be collected from lowest to highest concentrations. An effort should be made to collect samples in an upwind/upstream location and when site activities are at their lowest level.

A Field Blank is collected prior to collecting any samples to monitor ambient mercury levels and an Equipment Blank is collected to verify the equipment is free of contamination prior to the collection of a sample.

Field Blank Collection:

In an area expected or known to be free of high levels of mercury, the team will put on non-talc gloves and place plastic sheeting over the table or surface in which the sample collection is to be conducted. The team will then remove the bags containing the equipment (pump, flow-through cell meter, and/or water level meter) and containers from the coolers or storage containers in which they are packed. The team will remove the gloves and put on the Tyvek® or similar outer suit and new non-talc gloves.

The team members will perform the following procedures:

1 "Dirty Hands" opens cooler and takes out bagged sample container kit designated as the field blank bottle kit (filled with clean reagent water) and a bagged sample container that is empty. If a "Field Blank kit" is not available, proceed with bagged empty bottle kit)

2 "Dirty Hands" opens outer bag to field blank kit and empty bottle kit

3 "Clean Hands" opens inner bag of each and removes sample bottles

4 "Clean Hands" reseals inner bags

5 "Dirty Hands" reseals outer bags once the inner bag is sealed and sample bottle has been removed (by "Clean Hands")

6 "Dirty Hands" labels outer bag with sample identification information

7 "Clean Hands" removes caps from the bottle filled with reagent water and from the empty bottle and pours the clean reagent water into the empty bottle allowing no

headspace (if "Field Blank kit" is not available, rinse cap and bottle three times and fill bottle with clean reagent water from container supplied by the laboratory)

8 "Clean Hands" caps bottle, inverts bottle, and taps end to check for air bubbles

9 If air bubbles are present, the bottle is reopened and capped off with clean reagent water and recapped. This process is continued until no air bubbles are present.

10 "Dirty Hands" reopens outer bag so that "Clean Hands" can place sample bottle into inner bag

11 "Clean Hands" opens inner bag and places sample bottle into bag

12 "Clean Hands" reseals inner bag

13 After the inner bag with samples are sealed by "Clean Hands", "Dirty Hands" reseals outer bag.

14 "Dirty Hands" places bagged sample into cooler and closes cooler

15 "Dirty Hands" records information in the log book

16 The sampling team removes their gloves

Equipment Blank Collection:

Once the Field Blank sample is collected the team will collect an Equipment Blank sample. The team changes gloves. The following steps are to be performed if the sample tubing is to be decontaminated between locations. If new "clean" tubing is to be used at each location then proceed to step 8 and place clean tubing in Tub 3 and proceed as directed to collect the equipment blank sample.

Peristaltic Pump

The team members will perform the following procedures:

1. "Dirty Hands" prepares decontamination solutions and tubs

   - Tub 1 contains Alconox®/tap water solution
   - Tub 2 contains fresh tap water
   - Tub 3 contains reagent water
   - Tub 4 contains reagent water

2. "Dirty Hands" opens cooler and removes bag containing tubing kit and opens the outer bag

3. "Clean Hands" removes tubing from inner bag and places on pump taking care not to touch the pump housing or allow the tubing to touch the ground or other surfaces that may contaminate the tubing

4. "Dirty Hands" assists "Clean Hands" with tubing installation by touching the pump housing mechanism as appropriate and lowers tubing into Tub 1 (after "Clean Hands" has placed tubing on pump), hooks pump to battery, turns on controller and pumps 3 volumes of Alconox®/tap water solution through tubing

5. "Clean Hands" retrieves tubing from Tub 1 and places tubing in Tub 2

6. "Dirty Hands" turns on controller to pump fresh tap water from Tub 2 through tubing

7. After 3 volumes of tap water has been pumped through tubing, "Clean Hands" removes tubing from Tub 2 and places in Tub 3

8. "Dirty Hands" turns on controller to pump reagent water from Tub 3 through tubing

9. After 3 volumes of reagent water has been pumped through tubing, "Clean Hands" removes tubing from Tub 3 and places tubing in Tub 4

10. With tubing in Tub 4, "Dirty Hands" turns on controller and pumps 3 volumes of reagent water through tubing

11. Team members change gloves

12. "Dirty Hands" opens cooler and takes out bagged sample container

13. Dirty Hands" opens outer bag

14. "Clean Hands" opens inner bag and removes sample bottle

15. "Clean Hands" reseals inner bag

16. "Dirty Hands" reseals outer bag once the inner bag is sealed and sample bottle has been removed (by "Clean Hands")

17. "Dirty Hands" labels outer bag with sample identification information

18. "Clean Hands" removes cap and rinses cap and sample bottle with sample water three times

19. "Clean Hands" fills bottle with sample water allowing no headspace

20. "Clean Hands" caps bottle, inverts bottle, and taps end to check for air bubbles

21. If air bubbles are present, the bottle is reopened and capped off with clean reagent water and recapped. This process is continued until no air bubbles are present.

22. "Dirty Hands" reopens outer bag so that "Clean Hands" can place sample bottle into inner bag

23. "Clean Hands" opens inner bag and places sample bottle into inner bag
24. "Clean Hands" reseals inner bag
25. After the inner bag with samples are sealed by "Clean Hands", "Dirty Hands" reseals outer bag
26. "Dirty Hands" places bagged sample into cooler and closes cooler
27. "Dirty Hands" records information in the log book
28. "Clean Hands" removes water level meter and multi-parameter meter from storage bags, decontaminates meter(s) with solutions in Tubs 1,2, and 3 and places water level meter and multi-parameter meter into clean storage bags for transportation to sampling location
29. "Dirty Hands" places peristaltic pump into a storage bag to ready it for transportation to sampling location
30. The sampling team removes their gloves

Surface Water Sampling with a Peristaltic Pump

After collection of the Field Blank and Equipment Blank, the team proceeds to the sampling location. If sampling from a boat, both team members loads the boat with sampling equipment, places the boat in the water body to be sampled, and proceeds to the sampling location. The team changes gloves and places plastic sheeting over the boat surface in which the sample collection is to be conducted. The team changes gloves.

The team members will perform the following procedures:

1. "Dirty Hands" opens outer bag containing pump, battery, and bag containing Teflon® and SEBS resin tubing
2. "Clean Hands" removes tubing from inner bag and places in water to the desired depth
3. "Clean Hands" places tubing on pump taking care not to touch the pump housing or allow the tubing to touch the ground or other surfaces that may contaminate the tubing
4. "Dirty Hands" assists "Clean Hands" with tubing installation by touching the pump housing mechanism as appropriate and opens outer bag containing water level meter
5. "Clean Hands" removes water level meter from bag and places in water to the desired depth

6. "Clean Hands" connects multi-parameter meter flow through cell to pump outlet (if required)

7. "Dirty Hands" connects pump to battery, turns on controller and pumps sample to surface

8. "Dirty Hands" records water quality parameters (temperature, pH, specific conductance, turbidity, dissolved oxygen, salinity, and oxygen reduction potential) in field log book/forms

9. "Clean Hands" disconnects meter after water quality parameters are recorded

10. Team members change gloves

11. "Dirty Hands" opens cooler and takes out bagged sample container (if field duplicate or matrix spike/matrix spike duplicates are to be collected, two or three containers are required)

12. "Dirty Hands" opens outer bag

13. "Clean Hands" opens inner bag and removes sample bottle

14. "Clean Hands" reseals inner bag

15. "Dirty Hands" reseals outer bag once the inner bag is sealed and sample bottle has been removed (by "Clean Hands")

16. "Dirty Hands" labels outer bag with sample identification information

17. As sample water is flowing through pump, "Clean Hands" removes cap and rinses cap and bottles with sample water 3 times

18. "Clean Hands" fills bottle with sample water from tube allowing no headspace

19. "Clean Hands" caps bottle, inverts bottle, and taps end to check for air bubbles

20. If air bubbles are present, the bottle is reopened and capped off with clean reagent water and recapped. This process is continued until no air bubbles are present.

21. "Dirty Hands" reopens outer bag so that "Clean Hands" can place sample bottle into inner bag

22. "Clean Hands" opens inner bag and places sample bottle into inner bag

23. "Clean Hands" reseals inner bag and places it into outer bag

24. After the inner bag with samples are sealed by "Clean Hands", "Dirty Hands" reseals outer bag

25. "Dirty Hands" places bagged sample into cooler and closes cooler

26. "Dirty Hands" records information in the log book

27. "Clean Hands" removes equipment from sampling location and places equipment in bags for transportation

28. Team moves to decontamination area. The SEBS tubing is replaced prior to sampling each new location.

29. "Clean Hands" removes water level meter and multi-parameter meter from storage bags, decontaminates meter(s) with solutions in Tubs 1,2, and 3 and places water level meter and multi-parameter meter into clean storage bags

30. The sampling team removes their gloves

Groundwater Sampling with a Peristaltic Pump

After collection of the Field Blank and Equipment Blank, the team precedes to the sampling location. After the team arrives at the well, The team will the team will put on non-talc gloves and place plastic sheeting over the table or surface in which the sample collection is to be conducted. Nest, the team will remove the bags containing the equipment (pump, flow-through cell meter, and/or water level meter) and containers from the coolers or storage containers in which they are packed. The team changes gloves.

The team members will perform the following procedures:

1. "Dirty Hands" opens outer bag containing pump, battery, and bag containing Teflon® and SEBS resin tubing

2. "Clean Hands" removes tubing from inner bag and places in water to the desired depth

3. "Clean Hands" places tubing on pump taking care not to touch the pump housing or allow the tubing to touch the ground or other surfaces that may contaminate the tubing

4. "Dirty Hands" assists "Clean Hands" with tubing installation by touching the pump housing mechanism as appropriate and opens outer bag containing water level meter

5. "Clean Hands" removes water level meter from bag and places in water to the desired depth

6. "Clean Hands" connects multi-parameter meter flow through cell to pump outlet

7. "Dirty Hands" connects pump to battery, turns on controller and pumps sample to surface

8. "Dirty Hands" monitors water quality parameters (temperature, pH, specific conductance, turbidity, dissolved oxygen, and oxygen reduction potential) until stabilization is achieved (i.e. two consecutive measurements are within 5 percent and the water turbidity is less than 10 NTUs after three well volumes); records in field log book/forms

9. "Clean Hands" disconnects meter after stabilization

10. Team members change gloves
11. "Dirty Hands" opens cooler and takes out bagged sample container (if field duplicate or matrix spike/matrix spike duplicates are to be collected, two or three containers are required)
12. "Dirty Hands" opens outer bag
13. "Clean Hands" opens inner bag and removes sample bottle
14. "Clean Hands" reseals inner bag
15. "Dirty Hands" reseals outer bag once the inner bag is sealed and sample bottle has been removed (by "Clean Hands")
16. "Dirty Hands" labels outer bag with sample identification information
17. As sample water is flowing through pump, "Clean Hands" removes cap and rinses cap and bottles with sample water 3 times
18. "Clean Hands" fills bottle with sample water from tube allowing no headspace
19. "Clean Hands" caps bottle, inverts bottle, and taps end to check for air bubbles
20. If air bubbles are present, the bottle is reopened and capped off with clean reagent water and recapped. This process is continued until no air bubbles are present.
21. "Dirty Hands" reopens outer bag so that "Clean Hands" can place sample bottle into inner bag
22. "Clean Hands" opens inner bag and places sample bottle into bag
23. "Clean Hands" reseals inner bag
24. After the inner bag with samples are sealed by "Clean Hands", "Dirty Hands" reseals outer bag
25. "Dirty Hands" places bagged sample into cooler and closes cooler
26. "Dirty Hands" records information in the log book
27. "Clean Hands" removes equipment from sampling location and places equipment in bags for transportation
28. Team moves to decontamination area. The SEBS tubing is replaced prior to sampling each new location.
29. "Clean Hands" removes water level meter and multi-parameter meter from storage bags, decontaminates meter(s) with solutions in Tubs 1,2, and 3 and places water level meter and multi-parameter meter into clean storage bags
30. The sampling team removes their gloves

REFERENCES

Bloom. 1993.

USEPA Method 1631E

USEPA Method 1630 Draft

USEPA Method 1669

APPENDIX B

FIELD EQUIPMENT CALIBRATION PROCEDURES AND FIELD FORMS

APPENDIX B-1
YSI Field Calibration Form and Equipment Calibration Procedures

YSI CALIBRATION PRIOR TO SAMPLING

DATE ____/_____/___ TIME___:___:___
SONDE ID_____ HANDSET ID_____
BATTERY VOLTAGE_____

DISSOLVED OXYGEN
CHANGED DO MEMBRANE? YES NO If yes, when? ____/_____/___ ___:___:___
Note: If membrane is changed, wait 6 to 8 hours before completing DO test and final calibration
DO % VALUE BEFORE CALIBRATION _____%; AFTER CALIBRATION_____%

DO CHARGE _____(range 25 to 75) DO GAIN _____(range -0.7 to1.5)

CONDUCTIVITY
Note: Calibrate first to avoid carry-over from other standards (i.e. pH buffers are highly conductive)
CALIBRATION STANDARD USED_____µS/cm, TEMP_____°C
READING BEFORE CALIBRATION_____µS/cm, AFTER CALIBRATION_____µS/cm

CONDUCTIVITY CELL CONSTANT_____µS/cm (Range 5.0 ± 0.5)

pH
pH 7 VALUES BEFORE CALIBRATION: _____ (pH) AFTER CALIBRATION_____ (pH)

pH 7 MILLI-VOLT READINGS: _____mV Range -50 to +50 mV

ph 10 VALUES BEFORE CALIBRATION: _____ (pH) AFTER CALIBRATION_____ (pH)

pH 10 MILLI-VOLT READINGS: _____mV Range -130 to -230 mV

pH 4 VALUES BEFORE CALIBRATION: _____ (pH) AFTER CALIBRATION_____ (pH)

pH 4 MILLI-VOLT READINGS: _____mV Range 130 to 230 mV
Note: Span between pH 4 and 7, 7 and 10 mV numbers should be ~165-180 mV

REDOX POTENTIAL (ORP)
CALIBRATION STANDARD USED_____mV, CAL TEMP_____°C

READING BEFORE CALIBRATION_____mV, AFTER CALIBRATION_____mV

TURBIDITY
Wiper Parked ~180º from optics? Y N Note: Change wiper if probe is not parked correctly
TURBIDITY STANDARD _____ (NTUs)

VALUES BEFORE CALIBRATION: _____ (NTUs) AFTER CALIBRATION_____ (NTUs)

TURBIDITY STANDARD _____ (NTUs)

VALUES BEFORE CALIBRATION: _____ (NTUs) AFTER CALIBRATION_____ (NTUs)

CALIBRATION SUCCESSFUL? YES NO INITIAL_____

DESCRIBE ANY PROBLEMS ENCOUNTERED:_____
_____
_____

PREPARED BY/DATE: JP 07/13/04
CHECKED BY/DATE: AC 07/13/04

ATTACHMENT 4.1a-continued
YSI CALIBRATION CHECK AFTER SAMPLING

DATE ____/_____/___ TIME___:___:___
SONDE ID_____ HANDSET ID_____
BATTERY VOLTAGE_____

NOTE: CALIBRATION IS SUCCESSFUL WHEN THERE IS NO SIGNIFICANT DIFFERENCES (± 5 %) BETWEEN INITIAL CALIBRATION AND CALIBRATION CHECK

DISSOLVED OXYGEN
CHANGED DO MEMBRANE? YES NO If yes, when? ____/_____/___ ___:___:___
Note: If membrane is changed, wait 6 to 8 hours before completing DO test and final calibration
DO % VALUE BEFORE CALIBRATION_____%, AFTER CALIBRATION_____%
DO CHARGE _____ DO GAIN _____
CALIBRATION SUCCESSFUL? YES NO INITIAL_____

CONDUCTIVITY
Note :Calibrate first to avoid carry-over from other standards (i.e. pH buffers are highly conductive)
CALIBRATION STANDARD USED_____µS/cm, CAL TEMP_____°C
VALUE_____µS/cm
CONDUCTIVITY CELL CONSTANT_____µS/cm (Range 5.0 ± 0.5)
CALIBRATION SUCCESSFUL? YES NO INITIAL_____

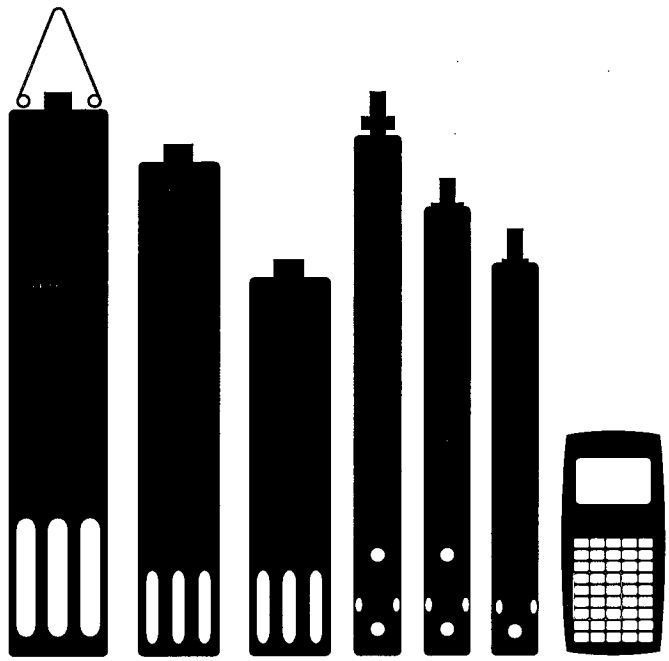
pH
pH 7 VALUE _____ (pH)
pH 7 MILLI-VOLT READINGS: _____mV Range -50 to +50 mV
ph 10 VALUE _____ (pH)
pH 10 MILLI-VOLT READINGS: _____mV Range -130 to -230 mV
pH 4 VALUE _____ (pH)
pH 4 MILLI-VOLT READINGS: _____mV Range 130 to 230 mV
Note: Span between pH 4 and 7, 7 and 10 mV numbers should be ~165-180 mV
CALIBRATION SUCCESSFUL? YES NO INITIAL_____

REDOX POTENTIAL (ORP)
CALIBRATION STANDARD USED_____mV, CAL TEMP_____°C
VALUE_____mV
CALIBRATION SUCCESSFUL? YES NO INITIAL_____

TURBIDITY
Wiper Parked ~180º from optics? Y N Note: Change wiper if probe is not parked correctly
TURBIDITY STANDARD 1 _____ (NTUs)
VALUE_____ (NTUs)
TURBIDITY STANDARD 2 _____ (NTUs)
VALUE _____ (NTUs)
CALIBRATION SUCCESSFUL? YES NO INITIAL_____

PREPARED BY/DATE: JP 07/13/04
CHECKED BY/DATE: AC 07/13/04

YSI *incorporated*



6-SERIES

6600 Sonde
6920 Sonde
6820 Sonde
600XLM Sonde
600XL Sonde
600R Sonde
610D Display/Logger
610DM Display/Logger

Environmental Monitoring Systems

YSI *incorporated*

1700/1725 Brannum Lane
Yellow Springs, Ohio 45387 USA
(800) 765-4974 (937) 767-7241
FAX: (937) 767-9320
Website: http://www.ysi.com
E-mail: info@ysi.com

Item # 069300
Drawing # A69300
Revision A
May 1999

```
----------------Logging---------------
1-Interval=00:15:00
2-Next at 07/17/96
3-Next at 18:00:00
4-Stop at 07/31/96
5-Stop at 18:00:00
6-File=clrlake3
7-Site=Clear Lake at Spillway
8-Bat volts: 9.0
9-Bat life 21.2 days
A-Free mem 18.9 days
B-Stop logging

Select option (0 for previous menu):
```

The display now shows the next date and time for logging and the stop date and time for the logging study. Most importantly, note that the bottom command now shows **B-Stop logging**, a confirmation that the logging has indeed been initiated.

The Unattended study will terminate when the duration you specified has expired or the batteries are expended. If you want to terminate sooner, simply select **2-Unattended** sample from the Run menu, then **B-Stop logging**. Select **1-Yes** and return to the Unattended setup menu.

```
Stop logging?
1-Yes
2-No

Select option (0 for previous menu):
```

2.9.2 CALIBRATE

All of the sonde sensors (except temperature) require periodic calibration to assure high performance. However, the calibration protocols for dissolved oxygen are significantly different depending on whether the sonde is being set up for spot sampling or longer term unattended monitoring studies. This difference is user-selectable and is required primarily because the optimal performance of the Rapid Pulse dissolved oxygen sensor cannot be attained unless the control of this sensor varies from short term to long term applications.

For spot sampling it is best to pulse the sensor continuously during the Run mode to attain the most accurate results and optimize the response time. However, this continuous pulsing is not ideal for longer term logging studies in which the sonde data is captured to sonde memory or to a data collection platform at much less frequent intervals (e.g. 15 minutes). Continuous pulsing not only shortens the time between required probe maintenance, but consumes more power. With

proper selection of the "Auto sleep" option (discussed in detail in **Section 2.9.8, Sonde Menu**), the user can configure the sonde software to either run continuously or "go to sleep" between samples to minimize DO probe wear and conserve power. The effect of this choice on the user interface relative to dissolved oxygen calibration is significant as described below:

- If "Auto sleep" is **deactivated**, the sonde runs continuously no matter what sample interval has been selected. Under these conditions, you retain manual control of the dissolved oxygen calibration routine, viewing the stabilization of the readings in real time and confirming the calibration with keyboard entries.

- If "Auto sleep" is **activated**, the sonde will 'warm up' the sensors for the period of time selected for the DO sensor. Under these conditions, you lose manual control of the DO calibration routine. DO will automatically calibrate after the selected time for warm up of the DO sensor has expired. In this mode of calibration, you do not observe stabilization of the readings in real time, but instead will observe a countdown of the warm up period followed by a message indicating that the DO calibration is complete.

Only the calibration of dissolved oxygen is affected by whether "Auto sleep" is on or off; the user retains manual control of the calibration of all other parameters regardless of the "Auto sleep" setting. Once warm up time has been utilized in DO calibration, the length of that time should not be changed during a study. A new calibration should be performed whenever the value of the warm up time is altered.

From the Main sonde menu select **2-Calibrate**. The Calibrate menu will be displayed. Only the enabled parameters will be available for calibration.

```
---------------Calibrate-------------
1-Conductivity       6-ISE3 NH4+
2-Dissolved Oxy      7-ISE4 NO3-
3-Pressure-Abs       8-ISE5 Cl-
4-ISE1 pH            9-Turbidity
5-ISE2 ORP           A-Chlorophyll

Select option (0 for previous menu):
```

CONDUCTIVITY

Select number **1 - Conductivity** to calibrate the conductivity probe and a second menu will offer you the options of calibrating in specific conductance, conductivity, or salinity. Calibrating any one option automatically calibrates the other two. After selecting the option of choice (specific conductance is normally recommended), you will be asked to enter the value of the standard used during calibration. Be certain that the units are correct. After pressing **Enter**, you will be able to follow the stabilization of the readings and confirm the calibration when the readings are stable by pressing **Enter** as instructed on the screen. Then, as instructed, press **Enter** again to return to the Calibrate menu.

DISSOLVED OXYGEN WITH AUTOSLEEP ON

If you intend to do Unattended Sampling, it is recommended that you turn Autosleep on and follow these instructions for DO calibration. If you intend to do Discrete Sampling, it is recommended that you turn Autosleep off and use the calibration instructions in the next section.

Select number **2 - Dissolved oxygen** to calibrate the oxygen probe. The submenu will offer you the option of calibrating in percent saturation or mg/L. After selecting the option of choice (percent saturation in water-saturated air is normally recommended), you will be prompted for the next step. Calibrating either of the choices will automatically calibrate the other.

For the percent saturation mode, be certain that the sensor has been thermally equilibrated in water-saturated air and that the sensor has stabilized prior to beginning the calibration routine, particularly after a membrane change. Relieve pressure in the cup if necessary.

Remember, the Calibration Cup is designed to be air-tight and must be loosened if it is used as a calibration chamber. See **Section 2.6, Calibration** for more details. Follow the screen prompt and enter the local barometric pressure in mm Hg, (inches Hg x 25.4), press **Enter**, and the calibration will automatically occur after the warm-up time which has been selected by the user (default is 40 seconds). Then, as instructed, press **Enter** again to return to the Calibrate menu.

For the mg/L mode, calibration is carried out in a water sample which has a known concentration of dissolved oxygen, usually determined by Winkler titration. For this calibration procedure, the sensor should be immersed in the water. After thermal equilibration, enter the known mg/L value, press **Enter**, and the calibration procedure will be carried out automatically as for the percent saturation mode above.

DISSOLVED OXYGEN WITH AUTOSLEEP OFF

If you intend to do Discrete Sampling, it is recommended that you turn Autosleep off and follow these instructions for DO calibration. If you intend to do Unattended Sampling, it is recommended that you turn Autosleep on and using the calibration instructions in the preceding section.

Select the **Dissolved Oxygen** option from the Calibrate menu to calibrate the oxygen probe. The submenu will offer you the option of calibrating in percent saturation or mg/L. After selecting the option of choice (percent saturation in water-saturated air is normally recommended), you will be prompted for the next step. Calibrating either of the choices will automatically calibrate the other.

For the percent saturation mode, be certain that the sensor has been thermally equilibrated in water-saturated air and that the sensor has stabilized prior to beginning the calibration routine, particularly after a membrane change. Relieve pressure in the cup if necessary. Remember, the Calibration Cup is designed to be air-tight and must be loosened if used as a calibration chamber. Then follow the screen prompt and enter the local barometric pressure in mm Hg, (inches Hg x 25.4), press **Enter**, and monitor the stabilization of the DO readings. After no changes occur for

approximately 30 seconds, press **Enter** to confirm the calibration. Then, as instructed, press Enter again to return to the **Calibrate** menu.

For the mg/L mode, calibration is carried out in a water sample which has a known concentration of dissolved oxygen, usually determined by a Winkler titration. For this calibration procedure, the sensor should be immersed in the water. After thermal equilibration, enter the known mg/L value, press **Enter**, and the calibration procedure will begin with similar viewing of stabilization and confirmation of calibration as for the percent saturation mode above.

NOTE: If you have resurfaced your DO sensor, we recommend running the probe continuously for 15-30 minutes or until good stability is realized. After a membrane change only, run the probe continuously for 3-4 minutes or until good stability is realized.

PRESSURE – ABS AND GAGE

Select number **3 - Pressure – Abs (non-vented) or Gage (vented)** to zero the depth sensor. The depth sensor is factory calibrated, but it is always necessary to zero the absolute sensor relative to the local barometric pressure. A minor correction is sometimes necessary to set Gage to exactly 0.000 feet. The zeroing procedure should be carried out with the sonde in air for this initial calibration. Alternatively, you may set zero or an offset while the sonde is submersed for "relative depth" applications. After the depth option is selected, enter 0.00 (or other appropriate number) at the prompt, press **Enter** and monitor the stabilization of the depth readings. After no changes occur for approximately 30 seconds, press **Enter** to confirm the calibration. As instructed, press **Enter** again to return to the Calibrate menu.

Zeroing the depth sensor by the above protocol (entering 0.00 at the screen prompt) will result in a measurement of the distance between the water surface and the ports of the depth module. In order for the observed depth readings to reflect the distance between the water surface and the actual probe array, measure the length between the upper hole and the bottom of the standard 6-inch sonde guard. Enter the length at the screen prompt instead of 0.00.

For best performance of depth measurements, users should ensure that the sonde's orientation remains constant while taking readings. This is especially important for vented level measurements and for sondes with side mounted pressure sensors.

pH

When selecting number **4 – ISE1-pH**, you will be given the choice of 1-point, 2-point, or 3-point calibrations.

Select the **1-point** option only if you are adjusting a previous calibration. If a 2-point or 3-point calibration has been performed previously, you can adjust the calibration by carrying out a one point calibration. Immerse the sonde in a buffer of known pH value and press **Enter.** You will be prompted to type in the pH value of the solution. Press **Enter** again, and the screen will display real-time readings that will allow you to determine when the pH and temperature readings have stabilized. Pressing **Enter** will confirm the calibration. Then, as instructed, press **Enter** again to return to the Calibrate menu. This calibration procedure adjusts only the pH offset and leaves the previously determined slope(s) unaltered.

Select the **2-point** option to calibrate the pH probe using only two calibration standards. In this procedure, the pH sensor is calibrated using a pH 7 buffer and pH 4 buffer. A two point calibration procedure (as opposed to a 3-point procedure) can save time if the pH of the media being monitored is known to be either basic or acidic. For example, if the pH of a pond is known to vary between 5.5 and 7, a two-point calibration with pH 7 and pH 4 buffers is appropriate. Three point calibration with an additional pH 10 buffer will not increase the accuracy of this measurement since the pH is not within this higher range.

To begin the calibration, immerse the sonde in one of the buffers and enter the actual pH value. Press **Enter**, and the screen will display real-time readings that will allow you to determine when the pH sensor has stabilized. Pressing **Enter** will confirm the calibration. Following the instructions on the screen, place the sonde in the second pH buffer, input the pH value, press **Enter**, and view the stabilization of the values on the screen in real time. After the readings have stabilized, press **Enter** to confirm the calibration. Then, as instructed, press **Enter** again to return to the Calibrate menu.

Select the **3-point** option to calibrate the pH probe using three calibration solutions. In this procedure, the pH sensor is calibrated with a pH 7 buffer and two additional buffers. The 3-point calibration method assures maximum accuracy when the pH of the media to be monitored cannot be anticipated. The procedure for this calibration is the same as for a 2-point calibration, but the software will prompt you to select a third pH buffer to complete the 3-point procedure.

ORP

Select number **5 - ISE2-ORP** to calibrate the ORP sensor. Immerse the sonde in a solution with a known oxidation reduction potential value (we recommend Zobell solution) and press **Enter.** You will be prompted to enter the ORP value of the solution. Press **Enter**, and monitor the stabilization of the ORP and temperature readings. After no changes occur for approximately 30 seconds, press **Enter** to confirm the calibration. Then, as instructed, press **Enter** again to return to the Calibrate menu.

The following calibrations are for the 6820, 6600 or 6920 sondes only. If you do not have one of these sondes, skip to Section 2.9.6, Report.

AMMONIUM

When selecting number **6 – ISE3-NH4+,** you will be given the choice of 1-point, 2-point, or 3-point calibrations for your ammonium (NH_4^+) sensor.

Select the **1-point** option only if you are adjusting a previous calibration. If a 2-point or 3-point calibration has been performed previously, you can adjust the calibration by doing a one point calibration. Immerse the sonde in any solution of known ammonium concentration and press **Enter.** You will be prompted to type in the NH_4^+ value (in mg/L of NH_4-N) of the solution you are using. Press **Enter** again, and the screen will display real-time readings which will allow you to determine when the NH4+ readings have stabilized. Pressing **Enter** will confirm the calibration.

Select the **2-point** option to calibrate the NH_4^+ probe using only two calibration standards which are both at approximately the temperature of your environmental sample. In this procedure, the NH_4^+ sensor is usually calibrated using solutions which contain 1 and 100 mg/L of NH_4-N . Be certain that the calibration solution and sensor are thermally equilibrated prior to entering NH_4^+ values.

To begin the calibration immerse the sonde in the 1 mg/L standard, press **Enter**, input the NH_4-N value, and again press **Enter.** The screen will display real-time readings which will allow you to determine when the sensor has stabilized. Pressing **Enter** will confirm the first calibration. Following the instructions on the screen, place the sonde in the second NH_4^+ standard, press **Enter**, input the correct concentration value, again press **Enter**, and view the stabilization of the values on the screen in real time. After the readings have stabilized, press **Enter** to confirm the calibration. Then, as instructed, press any key to return to the Calibrate menu.

Select the **3-point** option to calibrate the NH_4^+ probe using three calibration solutions, two at ambient temperature and one at a temperature significantly different from ambient. The 3-point calibration method should be used to assure maximum accuracy when the temperature of the media to be monitored cannot be anticipated. The procedure for this calibration is the same as for a 2-point calibration, but the software will prompt you to place the sonde in the additional solution to complete the 3-point procedure. Be certain that the calibration solution and sensor are thermally equilibrated prior to proceeding with the calibration. The recommended order of calibration standards is (1) 100 mg/L standard at ambient temperature, (2) 1 mg/L standard at ambient temperature, and (3) 1 mg/L standard at a different temperature (usually lower) than ambient. For best results, insure a temperature difference of at least 10 C°.

NITRATE

When selecting number **7 – ISE4-NO3**, you will be given the choice of 1-point, 2-point, or 3-point calibrations for your nitrate (NO_3^-) sensor. The procedure is identical to that for the ammonium sensor, except that the calibrant values are in mg/L of NO_3-N instead of NH_4-N.

CHLORIDE

When selecting number **8 – ISE5-CL-**, you will be given the choice of 1-point, 2-point, or 3-point calibrations for your chloride (Cl^-) sensor. The procedure is identical to that for the ammonium sensor, except that the calibrant values are in mg/L of Cl instead of NH_4-N. IMPORTANT: We recommend that the user employ standards for chloride that are 10 times greater than for ammonium and nitrate. Thus, the low calibration value should be 10 mg/L and the high calibration value should be 1000 mg/L Cl-. The difference is due to the fact that the effect of contamination of standards from inadvertent leakage of chloride ion from either the DO probe or the reference junction of the pH probe will be less significant at higher concentrations.

OPTIC TURBIDITY

When selecting number **9 – Turbidity,** there will be a choice of 1-point, 2-point, or 3-point calibrations for your turbidity sensor.

The **1-point** option is normally used to zero the turbidity probe in 0 NTU standard. Place the sonde in clear water with no suspended solids, and input 0 NTU at the screen prompt. Press **Enter** and the screen will display real-time readings that will allow you to determine when the turbidity readings have stabilized. Press **Enter** after the readings have stabilized to confirm the calibration and zero the sensor. Then, as instructed, press any key to return to the Calibrate menu. The 1-point option can also be used to adjust the turbidity system offset to any other turbidity value within the 0-1000 NTU range of the sensor while maintaining the slope(s) of previous 2- or 3-point calibration routines. For example, if desired, the sensor could be placed in 20 NTU standard, this value (rather than zero) Entered at the screen prompt, and the calibration confirmed to adjust the offset. The key to remember with regard to the 1-point calibration is that this procedure should only be used to update a previous 2-point or 3-point calibration.

Select the **2-point** option to calibrate the turbidity probe using only two calibration standards. In this case, one of the standards must be clear water (0 NTU) and the other should be in the range of known turbidity for the water to be monitored. For example, if the water to be evaluated is known to be low in turbidity, an appropriate choice of standards might be 0 and 10 NTU. However, for general purpose measurements an appropriate choice of standards is usually 0 and 100 NTU.

To begin the calibration, immerse the sonde in the 0 NTU standard, as instructed, and press **Enter.** It is mandatory that the 0 NTU standard be calibrated first. The screen will display real-time readings that will allow you to determine when the readings have stabilized. Pressing **Enter** will confirm the first calibration. Following the instructions on the screen, place the sonde in the second turbidity standard, input the correct turbidity value in NTU, press **Enter**, and view the stabilization of the values on the screen in real-time. After the readings have stabilized, press **Enter** to confirm the calibration. Then, as instructed, press any key to return to the Calibrate menu.

Select the **3-point** option for maximum accuracy over the entire range of 0 to 1000 NTU. As for the 2-point procedure, one of the standards must be 0 NTU. Because of the linearity characteristics of the sensors, we recommend that the other two standards have turbidity values of 10 and 100 NTU. However, the user can select any values that are deemed appropriate. The procedure for this calibration is the same as for a 2-point calibration, but the software will prompt you to place the sonde in the additional solution to complete the 3-point procedure.

For all turbidity calibration procedures, be certain that the standard and sensor are thermally equilibrated prior to proceeding with the calibration.

For further information related to setting up, calibrating and running turbidity measurements, refer to **Appendix E, Turbidity Measurements**.

OPTIC CHLOROPHYLL

When selecting number **Optic Chlorophyll,** there will be a choice of zeroing the full scale range of the fluorescence sensor (**Fluor Zero**), or calibrating with actual chlorophyll standards (**µg/L 1-point, 2-point, or 3-point**).

If you have selected **Fluor %FS** as a parameter in Report Setup, the sonde will report only relative values of fluorescence in the sample being measured. These values could then be

converted into actual chlorophyll concentrations in µg/L by using a post-calibration procedure, after the chlorophyll content of grab-samples taken during a sampling or monitoring study has been analyzed in a laboratory. This determination can involve conducting the extractive analysis procedure described for chlorophyll in *Methods for the Examination of Water and Wastewater* or by carrying out an *in situ* measurement of chlorophyll using a commercial benchtop fluorometer.

The **Fluor zero** option is used to zero the fluorescence probe in a medium that is chlorophyll-free. Place the sonde in clear water, and input 0 at the screen prompt. Press **Enter** and the screen will display real-time readings that will allow you to determine when the fluorescence readings have stabilized. Press **Enter** after the readings have stabilized to confirm the calibration and zero the sensor. Then, press any key to return to the Calibrate menu.

If you select **Chl µg/L** in the initial calibration routine, there will be a choice of 1-point, 2-point, or 3-point options. The 1-point selection is normally used to zero the fluorescence probe in a medium that is chlorophyll-free. If you use this method, you will either choose to utilize the default sensitivity for chlorophyll in the sonde software or to update a previous multipoint calibration. Usually you will place the sonde in clear water, and input 0 µg/L at the screen prompt. After pressing **Enter** the screen will display real-time readings allowing you to determine when the chlorophyll readings have stabilized. Press **Enter** after the readings have stabilized to confirm the calibration and zero the sensor. Then, as instructed, press any key to return to the Calibrate menu.

The 1-point option can also be used to adjust the chlorophyll system offset to any other chlorophyll value within the 0-200 µg/L range of the sensor while maintaining the slope(s) of previous 2- or 3-point calibration routines. For example, the sensor could be placed in 20 µg/L standard and this value (rather than zero) entered at the screen prompt, and the calibration confirmed to adjust the offset. The 1-point calibration procedure should only be used to update a previous 2-point or 3-point calibration or to accept the limitations of the default sensitivity.

Note: For the 2-point and 3-point calibrations described below, standards of known fluorescence are required. Two general types of standards can be used: (a) phytoplankton suspensions of known chlorophyll content, and (b) dye solutions whose fluorescence can be correlated to that of chlorophyll. The user is responsible for determining the chlorophyll content of phytoplankton suspensions, either by employing the extractive analysis procedure described in *Standard Methods for the Examination of Water and Wastewater,* or by analyzing the suspension *in situ* using a laboratory fluorometer. See Section 5, **Principles of Operation** and **Appendix I, Chlorophyll** of this manual for more information about chlorophyll standards.

Select the **2-point** option to calibrate the chlorophyll probe using only two calibration standards. In this case, one of the standards must be clear water (0 µg/L) and the other should be in the range of a known chlorophyll content of the water to be monitored. For example, if the water to be evaluated is known to be low in chlorophyll, an appropriate choice of standards might be 0 and 10 µg/L. However, for general-purpose measurements an appropriate choice of standards is usually 0 approximately 150 µg/L.

To begin the calibration, immerse the sonde in the 0 µg/L standard, as instructed, and press **Enter.** It is mandatory that the 0 µg/L standard be calibrated first. The screen will display real-time readings that will allow you to determine when the readings have stabilized. Pressing

Enter will confirm the first calibration. Following the instructions on the screen, place the sonde in the second chlorophyll standard, input the correct value in µg/L, press **Enter**, and view the stabilization of the values on the screen in real-time. After the readings have stabilized, press **Enter** to confirm the calibration. Then, as instructed, press any key to return to the Calibrate menu.

Select the **3-point** option for maximum accuracy over the entire range of 0 to 400 µg/L. As with the 2-point procedure, one of the standards must be 0 µg/L. The user can select any values for the second and third standards that are deemed appropriate. The procedure for this calibration is the same as for a 2-point calibration, but the software will prompt you to place the sonde in the additional solution to complete the 3-point procedure.

For all chlorophyll calibration procedures, be certain that the standard and sensor are thermally equilibrated prior to proceeding with the calibration

For further information related to calibrating your chlorophyll sensor, refer to Section 5, **Principles of Operation** and **Appendix I, Chlorophyll** of this manual.

2.9.3 FILE

Selections from the File menu allow the user to access data that has been stored in the sonde flash disk memory. Select number **3-File** from the Main menu.

```
------------------File-----------------
1-Directory      4-View file
2-Upload         5-Quick view file
3-Quick Upload   6-Delete all files


Select option (0 for previous menu): 1
```

- Select number **1-Directory** to view all files currently stored in sonde flash disk memory. The screen below shows 5 files that suggest the sonde was used to spot check two bridge locations, deployed for a week in the lake (sampling every 15 minutes), then used to recheck the bridge locations.

APPENDIX B-2
Chain-of Custody Form

OLIN CORPORATION

CHAIN OF CUSTODY RECORD

PROJ. NO.	PROJECT NAME
SAMPLERS: *(Signature)*	

ANALYSES REQUIRED

Instructions: List samples individually. Write time in military time. Record sample type as water, soil, etc. and as grab or composite. Record special handling instructions or precautions in remarks section. Record method of shipment in comments section.

Station or Sample No.	Date	Time	Sample Type	Number Of Containers							Remarks

Relinquished by: *(Signature)*	Date/Time	Received by: *(Signature)*	Relinquished by: *(Signature)*	Date/Time	Received by: *(Signature)*
Relinquished by: *(Signature)*	Date/Time	Received by: *(Signature)*	Relinquished by: *(Signature)*	Date/Time	Received by: *(Signature)*
Relinquished by: *(Signature)*	Date/Time	Received for Laboratory by: *(Signature)*	Date/Time	Comments	

DISTRIBUTION: Original and Pink copies accompany sample shipment to laboratory. Pink copy retained by laboratory. Yellow copy retained by samplers.

APPENDIX B-3
Example Sample Label

EXAMPLE SAMPLE LABEL

Site: Olin McIntosh OU-2
Sample ID#: **OU2B-SW-101DS-08**
Sample Location ID: B-1
Matrix: Surface Water
Analysis: Total Low-Level Mercury by EPA 1631E
Container: 500 mL Teflon
Preservative: No Headspace/Cool to 4^{o}C/lab pres. w/Bromine monochloride
Project #: 6100-08-0035
Date:_____ Time:_____
Initials:_____

APPENDIX C

DATA VALIDATION CHECKLIST

Client: **Olin** Project: **McIntosh** Sampling Event:_____

Laboratory: _____ Rpt. Number: _____ Rpt. Date: _____

	Laboratory Data Review Checklist	**Yes**	**No**	**Not Applicable**
1.	Laboratory analytical data report appears complete (all data results present for all samples submitted for analysis), and there are no apparent transcription errors.	___	___	___
2.	Samples analyzed within applicable holding times.	___	___	___
3.	Trip blanks, field blanks, equipment blanks, and/or laboratory method blanks are free of contamination.	___	___	___
4.	If field duplicate samples collected, Relative Percent Difference (RPD) is less than 35% for water and 50% for soil/sediment/tissue.	___	___	___
5.	Surrogate recoveries (organic analyses only) are within laboratory reported recovery acceptance ranges.	___	___	___
6.	If Matrix Spike/Matrix Spike Duplicate samples required to meet project objectives, recoveries and RPD within laboratory reported acceptance ranges.	___	___	___
7.	Reported detection limits meet project objectives and are capable of achieving applicable site standards.	___	___	___
8.	Completed Chain-of-Custody received noting sample/custody seal condition.	___	___	___
9.	Instrument tune, initial calibration, calibration verification, and continuing calibration within method criteria.	___	___	___
10.	Internal standard responses and retention times (where applicable) are within laboratory reported acceptance ranges.	___	___	___
11.	Serial dilutions and post digestion spikes (metals only) are within reported acceptance ranges.	___	___	___
12.	Column breakdown and/or second column confirmation (where applicable) are within method acceptance limits.	___	___	___
13.	Report uploaded to Centric Project.	___	___	___
14.	DQE sheet uploaded to Centric Project.	___	___	___

COMMENTS:

CHECKED BY: _____ DATE: _____ rev. 01 6/5/08